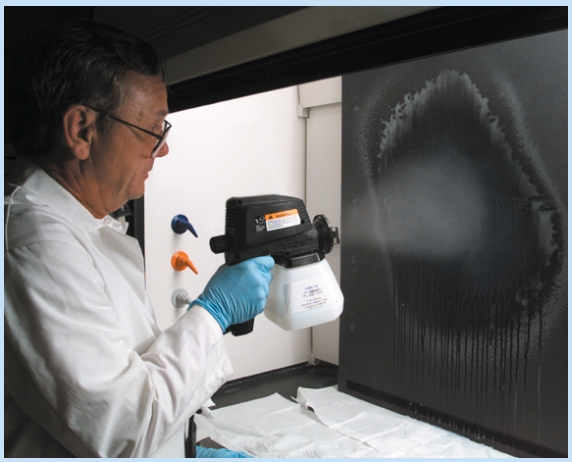
UCRL-AR-143212

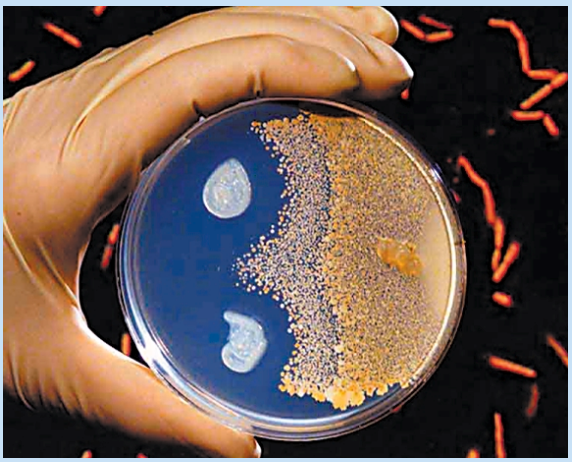
Oxidative Decontamination of Chemical and Biological Agents Using L-Gel

Raymond McGuire and Ellen Raber

Co-Investigators: D. Mark Hoffman, Paula Krauter, Donald Shepley, Armando Alcaraz, Emilio Garcia, Jeff Elliot, and Tina Carlsen

July, 2001





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This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

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Executive Summary

Developing a decontaminating agent for application to civilian facilities in response to a terrorist incident or catastrophic release poses many challenges. Our principal focus was to address the requirement that a reagent be not only effective against both chemical and biological warfare agents but also environmentally friendly. We have developed such a formulation by gelling an aqueous solution of a mild commercial oxidizer, Oxone™, with a commercial fumed silica gelling agent, Cab-O-Sil EH-5. The system has been named L-Gel.

L-Gel can be premixed and has an effective shelf life of at least one year. The system is thixotropic, and after liquefaction by manual or mechanical stirring, it can be applied using essentially any type of commercially available spray device with about any type of stainless-steel atomizing nozzle. L-Gel clings to walls and ceilings and does not harm carpets or painted surfaces.

L-Gel has been independently tested against all classes of chemical warfare agents and against various biological warfare agent surrogates, including spore-forming bacteria, and nonvirulent strains of real biological agents. The results of several years of such testing are summarized in this report. The data show that L-Gel is as effective against chemical agents and biological materials, including spores, as the best military decontaminants.

1. Introduction

Most previous efforts to develop decontaminating agents for chemical warfare (CW) agents have focused on militarily important scenarios and on hydrolysis as the principal reaction (McGuire et al., 1999a, 1999b). Our work at Lawrence Livermore National Laboratory (LLNL) and related field testing at four different offsite facilities explores other mechanisms, namely oxidation, and focuses on the decontamination of civilian facilities. We attempted to develop a single reagent and an acceptable decontamination method that is effective against all threats, including both CW agents and biological warfare (BW) agents (Krauter and Garcia, 2000; McGuire et al., 1999c; Raber et al, 2000).

The requirements for decontamination in the civilian sector are not only demanding but can also be somewhat different than decontamination requirements from a military perspective (Raber, 2000). Current military decontamination techniques aimed at CW agents are corrosive and can cause considerable collateral damage to military and civilian facilities and equipment. For this reason, more environmentally friendly decontaminants are of interest for all applications.

At the outset of our work, there were no effective decontamination tools that could be used in a high-tech environment, such as a computerized office or airplane cockpit, that would not destroy the electronics. To address potential settings, and to help direct our thinking about acceptable types of decontamination systems, we considered three types of civilian scenarios in which an incident could potentially occur. They are an outdoor scenario such as a stadium, a semi-enclosed scenario such as a subway, and an enclosed scenario such as an office. Methods for use on interior surfaces can have quite different requirements than those appropriate for outdoor use, where natural attenuation over time might be adequate in certain cases.

The military requires very fast action, whereas decontamination times on the order of several hours may be enough for the civilian sector. Rather than speed, considerations that are more important in a civilian scenario include availability of a reagent, ease of application, minimal training, easy deployment by a variety of dispersal mechanisms, and acceptable expense.

In some cases, the military needs decontamination technologies that essentially allow personnel to continue with a battle. In other cases, military requirements are not dissimilar to civilian requirements. Civilian facilities will, of necessity, be reoccupied for long periods without protective equipment. Exposures to environmentally hazardous material (e.g., carcinogens) and long-term health consequences to civilian populations, including susceptible individuals such as pregnant women or the immunocompromised, are major concerns. Civilian decontamination must seek to minimize adverse health effects, address relevant social and political issues, and be defensible to both regulatory agencies and the public.

Finally, the level of decontamination required influences the choice of decontamination systems. Understanding and influencing the answer to the question: “How clean is clean enough?” is key (Raber, 1999; Raber et al., 2001). Methods that destroy 90% of a threat are not effective if the objective is to destroy 99.999999% of the same agent. Similarly, it would be deceiving to imply that 100% of an agent is destroyed if the level of detection during sampling and analysis is only one part in a hundred thousand. Whereas future studies need to resolve all these issues, our work focused on the immediate problem of developing an effective decontamination reagent and application method that can be currently deployed.

1.1 Requirements and Goals

Several considerations drove our research and development efforts. To the extent possible, we worked to address the following optimal requirements for a decontamination system that can be used in civilian settings:

- **Time considerations**—Facility decontamination would occur after civilian individuals are evacuated, and decontamination of a site should not exceed three days.

- **Noncorrosive**—The system to be used for decontamination should be noncorrosive, result in minimal collateral damage, and protect high-value equipment.

- **Nontoxic**—The system itself should be nontoxic, and its application should result in the detoxification and/or decomposition of CW and BW agents into environmentally acceptable byproducts.

- **Ease of use**—Use of the system should require uncomplicated equipment and minimal training.

- **Deployability**—The system should be easily deployable by various dispersal or application methods and allow for use in different scenarios (outdoor, semi-enclosed, or enclosed).

From the foregoing considerations, our work focused on achieving the following goals:

- First and foremost, to develop a single nontoxic and noncorrosive decontamination system for all CW and BW agents.

- To evaluate various reagents for effective detoxification and/or degradation of CW and BW agents to nontoxic and environmentally acceptable components, rather than complete destruction of contaminant(s).

- To develop an easy-to-use decontamination system for use by first responders as well as specialized decontamination teams.

- To address the issues of public perception and stakeholder acceptance, including collateral damage and recertification, which are paramount in a domestic, urban scenario.

1.2 Rationale for Focus on the Oxidative Approach

Hydrolysis in basic media works well for the G-type chemical agents, and much previous effort has been put into kinetic and mechanistic studies. The G agents include Tabun (GA), Sarin (GB), and Soman (GD), among others. The reaction proceeds by a bimolecular nucleophilic substitution (SN2) mechanism, with the hydroxyl ion as the attacking species and fluoride or, in the case of GA, cyanide ion as the leaving group.

Hydrolysis works less well with Sulfur mustard (H or HD) because of the low aqueous solubility of mustard and its propensity to form protective micelles that effectively impede the reaction. If very dilute solutions and high agitation are used, then hydrolysis proceeds rapidly. Other work has demonstrated the effectiveness of prior oxidation of sulfur to form the sulfoxide or sulfone followed by hydrolysis of the carbon–chlorine (C–Cl) bond. A co-solvent system (aqueous + miscible organic) is the medium of choice.

In contrast, direct base hydrolysis is not effective for V agents, an example of which is VX. However, oxidation of the sulfur in VX in aqueous acid medium is rapidly followed by hydrolysis to nontoxic products. An acidic medium also causes protonation of the amine nitrogen, both increasing the solubility of VX and enhancing the oxidation of sulfur.

Based on the foregoing considerations for CW agents, we focused initially on aqueous acidic oxidation with simultaneous hydrolysis of the contaminant agent to achieve decontamination. The choice of oxidation as an approach to the detoxification of chemical agents is a result, in large part, of work performed over a long period of time at the Edgewood Chemical and Biological Center (ECBC). We also investigated the system for its effectiveness in destroying live biological agent organisms and spores.

We chose an acidic solution of oxidizer primarily to protonate the amine nitrogen in VX to a quaternary ammonium ion. This approach serves two functions. First, it increases the solubility of VX in the aqueous medium. Second, it blocks the nitrogen to oxidation. The oxidation then occurs preferentially on the sulfur. Subsequent cleavage of the phosphorous–sulfur (P–S) bond easily occurs by hydrolysis, and the VX is destroyed without the formation of toxic byproducts. The oxidation of sulfur in mustard followed by the hydrolysis of the C–Cl bond to the nontoxic sulfoxide and sulfone diglycols also takes place in an acidic medium. Detoxification of G agents using the same reagents presents the greatest challenge.

Whereas G agents hydrolyze in acid media, the rate curve as a function of pH goes through a minimum at approximately the pH of the oxidizing solution (pH = 2). A small amount of Cu^{2+} was added in an attempt to catalyze the G agent hydrolysis. As will be shown, the added Cu^{2+} did not yield the desired effect. However, a gelled oxidizing solution of Oxone™ did effectively destroy the G agent surrogate, and in subsequent experiments, GD itself.

Acidic oxidation is also an effective approach for the detoxification of biological agents. As will be discussed in more detail, certain BW agents, especially bacterial spores, are extremely difficult to kill. A strong oxidizer in a low-pH solution (<2) oxidizes the thiol groups in proteins and enzymes. It also forms free hydroxyl radicals, which can cause DNA and RNA strand breakage. Thus, a strong oxidizer includes two mechanisms to destroy BW agents.

1.3 Use of CW Surrogates

All of the work performed at LLNL on decontamination of CW agents must be done using surrogates rather than the real CW agents themselves. Surrogates were selected on the basis of several considerations. A CW surrogate must replicate as closely as possible those properties of the real CW agent that are important to decontamination efforts. Surrogates must contain the moieties of interest along with specific functional chemical groups. However, even though decontamination requires the scission of particular chemical bonds, total destruction of the toxic molecule is neither necessary nor cost effective. Figure 1 shows the selected chemical agent surrogates for use in oxidation/hydrolysis experiments conducted at LLNL. Real CW agents were subsequently used in field studies conducted offsite.

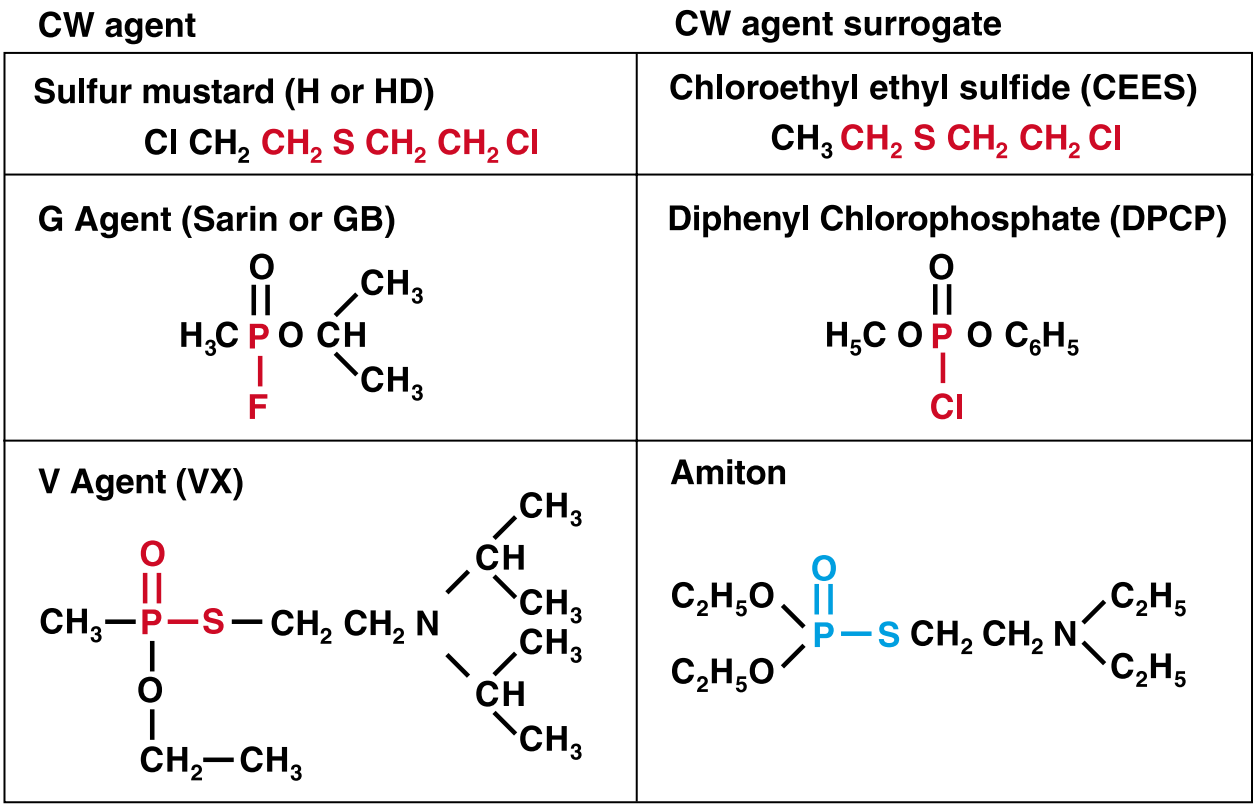

Figure 1. Real chemical agents and chemical agent surrogates.

Although the surrogates do not exactly mimic the rates of reaction of real CW agents, they undergo oxidation and/or hydrolysis within a factor of 1 or 2 of the rate of the agents they replicate; that is, the differences are not by orders of magnitude. The availability of Amiton for use as a surrogate for VX is particularly useful in that it, like VX, contains O=P–S bonds as well as the amine nitrogen two carbons removed from the sulfur. Thus, the chemistry of VX is well reproduced.

1.4 Use of BW Surrogates

All laboratory and field testing related to the decontamination of BW agents must be done using surrogates. Samples of real BW agent strains are confined in and limited to Biosafety Level 4 facilities. Surrogates were selected to replicate as closely as possible those properties of the real BW agents that are important to decontamination, and to meet Biosafety Level 1 or 2. We used *Bacillis subtilis* var. *niger*, also known as *Bacillus globigii* (BG), which is a surrogate for spore-forming agents such as anthrax. *Pantoea herbicola* (formerly known as *Erwinia herbicola*) was the surrogate for vegetative, nonspore-forming bacterial agents such a plague. Ovalbumin was the surrogate protein for Botulinum toxin. Table 1 shows the selected biological agent surrogates for use in many of our oxidation/hydrolysis experiments.

Table 1. Real biological agents and biological agent surrogates.

Biological agent	Biological agent surrogate meeting Biosafety Level 1
Anthrax	*Bacillus subtilus* var. *niger* (BG) spores
	• Gram positive and spore-forming bacteria
	• Durable spore common in certain soils, nonvirulent
	• Easily grown in culture, easily detected
Plague (Bubonic)	*Pantoea herbicola*
	• Vegetative, nonspore forming, gram-negative bacteria
	• Nonvirulent, found on plant leaves
	• Easily grown in culture, easily detected
Botulinum toxin	Ovalbumin (tested in gels only, not in field tests)
	• High-molecular-weight protein
	• Benign and nontoxic

The decontamination of BW agents requires cellular and protein destruction, as well as DNA or RNA destruction. Our selection of BW surrogates included BG spores because spores are far more difficult to kill than vegetative cells. Figure 2 shows the inner membrane, cortex, coats, and exosporium around the core of a bacterial spore. Even though BG closely simulates the actual BW agent, *Bacillus anthracis*, the surrogate is easier to kill than the real agent.

In a subsequent series of tests conducted during 2000, we obtained approval to use strains of the real biological agents *Bacillus anthracis* (Sterne) and *Yersinia pestis* (strain D27). These Biosafety Level 2 strains are rendered nonvirulent and can be experimentally studied because their complete genome is known, and they do not contain the toxic plasmids that are present in the real BW agents.

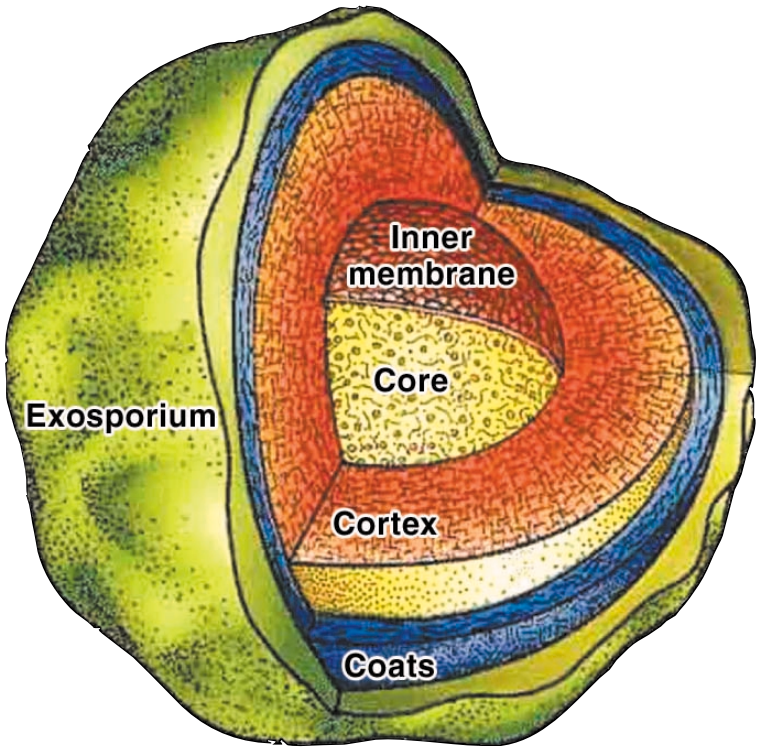

Figure 2. Cross section of a bacterial spore. (Drawing after J. Pettigrew.)

2. Evaluation of Carriers, Oxidants, and Application Systems

Because the focus of our overall effort was on the decontamination of civilian facilities, including building interiors, spraying only water-based solutions of decontaminants may not be effective in all cases. Consequently, we investigated carriers that would increase the contact time between the contaminant and decontaminating agent and would cling to walls and ceilings. The two carrier systems we assessed are aqueous foams and gelled aqueous solutions.

Several available decontaminating agents are effective against either CW or BW agents. Examples of effective reagents or mechanisms include a 5% solution of sodium hypochlorite, paraformaldehyde, or energy sources such as incineration and x rays. However, problems such as corrosivity, carcinogenicity, and collateral damage preclude their use in civilian scenarios. Our focus was on the selection of an effective decontaminating agent and application system that is safe for human use, nondamaging to commonly used materials and surfaces, and environmentally benign.

2.1 Aqueous Foams

Initial work in the area of aqueous foams involved materials proposed by Sandia National Laboratories. The two foam formulations provided are identified as anionic foam AFC-380 and cationic foam 1531-13-2. The exact formulation of the two foams is considered to be Proprietary Information by Sandia. In general terms, the anionic foam contains an anionic surfactant, long- and short-chain alcohols, a glycol-ether, other constituents, and is >95% water. The cationic foam contains various organic ammonium salts, high-molecular-weight polyols, long-chain alcohols, other constituents, and is >90% water.

The results of experiments on the foam formulations showed conclusively that organic-based foam components would not have long-term compatibility with oxidizing agents. Appendix A provides details on the aqueous foam experiments.

2.2 Gels

Given the compatibility problems with common organic foams, other potential carriers were explored. Gels of colloidal silica were evaluated as thickening agents for the oxidizer solutions proposed for decontamination of chemical and biological agents. LLNL has extensive experience with colloidal silica in silicone rubbers, perfluorinated solvents, and explosive formulations. For example, from the late 1960s to the late 1980s, LLNL researchers developed a series of extrudable high explosives based on the gelling of polar energetic liquids. Even though this research did not advance to the production stage, the development served as an experience base for formulation, characterization, and dispersal system design and fabrication. It was a logical step, therefore, to adapt this work to the gelling of aqueous oxidizers for candidate chemical and biological agent decontaminants.

Gelation using fumed silica was chosen for several reasons.

1. Because of their thixotropic nature, gels tend not to sag or flow down walls or off ceilings, thereby increasing the concentration of active ingredient, oxidizing agent, or other constituent in the area where it can do the most good.

2. Silicon dioxide colloidal particles are commercially available and do not require any special facility to prepare.

3. The inert characteristics of the particles, compared to carbon blacks or other colloidal particles, allow them to survive in the strong oxidant solutions currently used or proposed for decontamination of various agents.

4. Such gels lend themselves to simple delivery systems, such as Simplex sprayers or air-assisted sprayers.

5. Such gels may, because of the surface characteristics of fumed silica, be able to absorb certain of the chemical or biological agents (or embed spores into the gel) or to catalyze the decomposition of certain chemical agents.

6. Once the decontamination process is complete, such gels can easily be cleaned off surfaces or buildings by vacuum.

If the carrier liquid is water or a volatile organic, it will evaporate within several hours, increasing the concentration of solid oxidant. Once the liquid has evaporated, the weak silica gel residue, unreacted oxidizer, and entrapped decontaminated agents can be vacuumed and disposed of as nonhazardous waste. Although the amorphous silica is not chemically hazardous and does not represent an inhalation hazard, it would be expedient to remove the residual material before allowing personnel to re-enter facilities. For outdoor application, no cleanup is necessary. U.S. EPA-certified laboratory analysis (using methods 8260/8270 for volatiles and semi-volatiles) of the reacted material show the residual byproducts to be nonhazardous.

The various gelling agents that were evaluated are:

• Cab-O-Sil EH-5 • Aerosil 200 • COK 85 • Alumina C • Hi-Sil T-700.

Appendix B provides details on development of gelled aqueous oxidizer formulations leading to selection of Cab-O-Sil EH-5 fumed silica as a gelling agent.

2.3 Laboratory Testing of Oxidants

2.3.1 Initial Lab Screening

A variety of oxidants were initially evaluated in the laboratory against CW surrogates (DPCP, CEES, and Amiton) and then against BW surrogates (spores, vegetative bacteria, and ovalbumin). Table 2 lists the oxidants that were tested, and Appendix C discusses the testing procedures and evaluations of oxidizing agents in more detail. In general, experimental testing was conducted in triplicate with controls and blanks. Not all oxidants were tested against all CW and BW surrogates, but from 7 to 9 oxidants were generally tested against most surrogates and evaluated for efficacy.

Table 2. Oxidants evaluated against CW and BW surrogates.

Sodium hypochlorite (positive control)
No treatment (negative control)
Hydrogen peroxide
Potassium permanganate
Classic Fenton's reagent (3% hydrogen peroxide, 10 ppm $CuSO_4$ @ pH = 3)
LANL Fenton's reagent (5% $CuCl_2$, 1% ascorbic acid, KCl and HCl buffer @ pH = 2)
Cupric chloride (5%)
Peroxydisulfate
DOW™ liquid (foam) bathroom cleaner
Virkon S™
Potassium peroxymonosulfate + copper ion (10 ppm $CuSO_4$)
Potassium peroxymonosulfate + surfactant (Snoop™)
Potassium peroxymonosulfate (Oxone™)

As shown in Figure 3, the initial laboratory oxidation experiments on CW surrogates demonstrated that potassium peroxymonosulfate effectively destroys Amiton (99+% oxidized after 40 min) and CEES (100% oxidized after 30 min). Initial testing of the G agent surrogate DPCP was unsuccessful and is described in more detail in Appendix C.

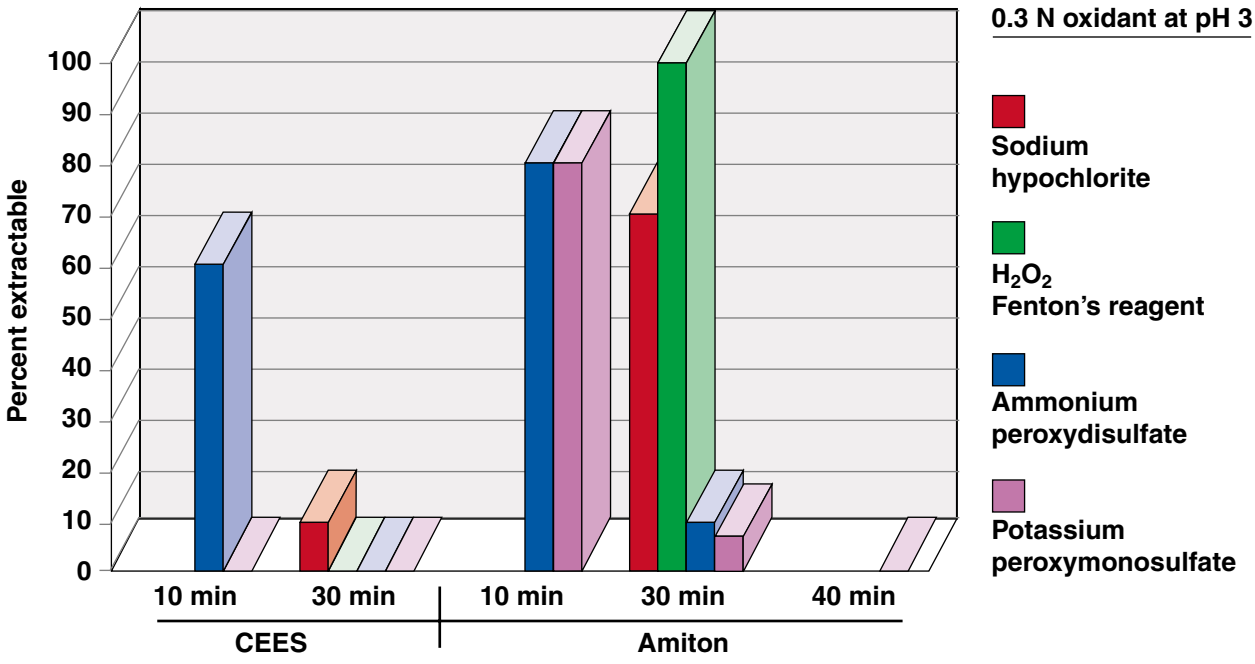

Figure 3. Oxidation of CEES and Amiton by four oxidants.

Figure 4 summarizes the results of two years of laboratory tests to assess the concentration dependency of several oxidizing agents against BG spore survival after 30-min exposures. The goal in this evaluation was to find the most effective decontaminating agent at the lowest effective concentration. The concentrations evaluated for biocidal effect of peroxymonosulfate ranged from 0.01 to 1.0 N. The BG spore culture was diluted to approximately 5×10^8 cells/mL for each of the tests, plating and incubation at 30°C were done in nutrient agar media, and colonies were visually counted (see Appendix C for more detail on these and related tests). Potassium peroxymonosulfate was 99+% effective at a concentration of 1.0 N. In addition, we found that iron and copper catalysts had no effect on the biocidal activity of peroxymonosulfate on BG spores.

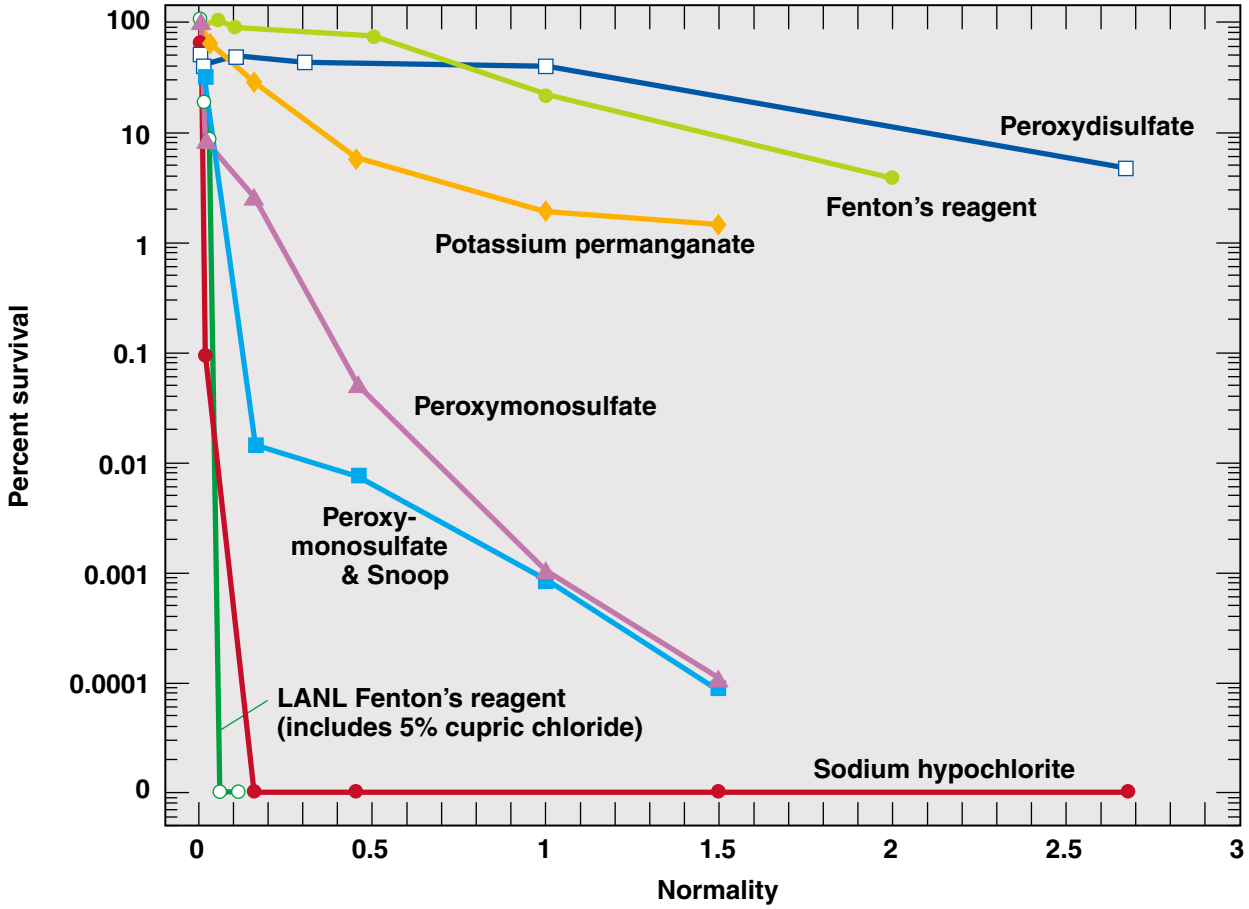

Figure 4. Percent survival of *B. subtilis* var. *niger* (BG) spores versus concentration of various oxidizing agents.

BG spores were tested for damage to spore wall structure after 30-min exposure to several acid and alkaline biocides at 22°C. Figure 5 shows spores stained with malachite green and safranin (red) at a magnification of 1,600×. The safranin dye penetrates only spores with damaged spore walls. Both acid and alkaline biocides damaged the spore wall structure, and biocides containing detergents showed cell clumping. Clumping is an undesirable effect because it becomes more difficult for a decontaminating agent to reach interior areas of a cell cluster.

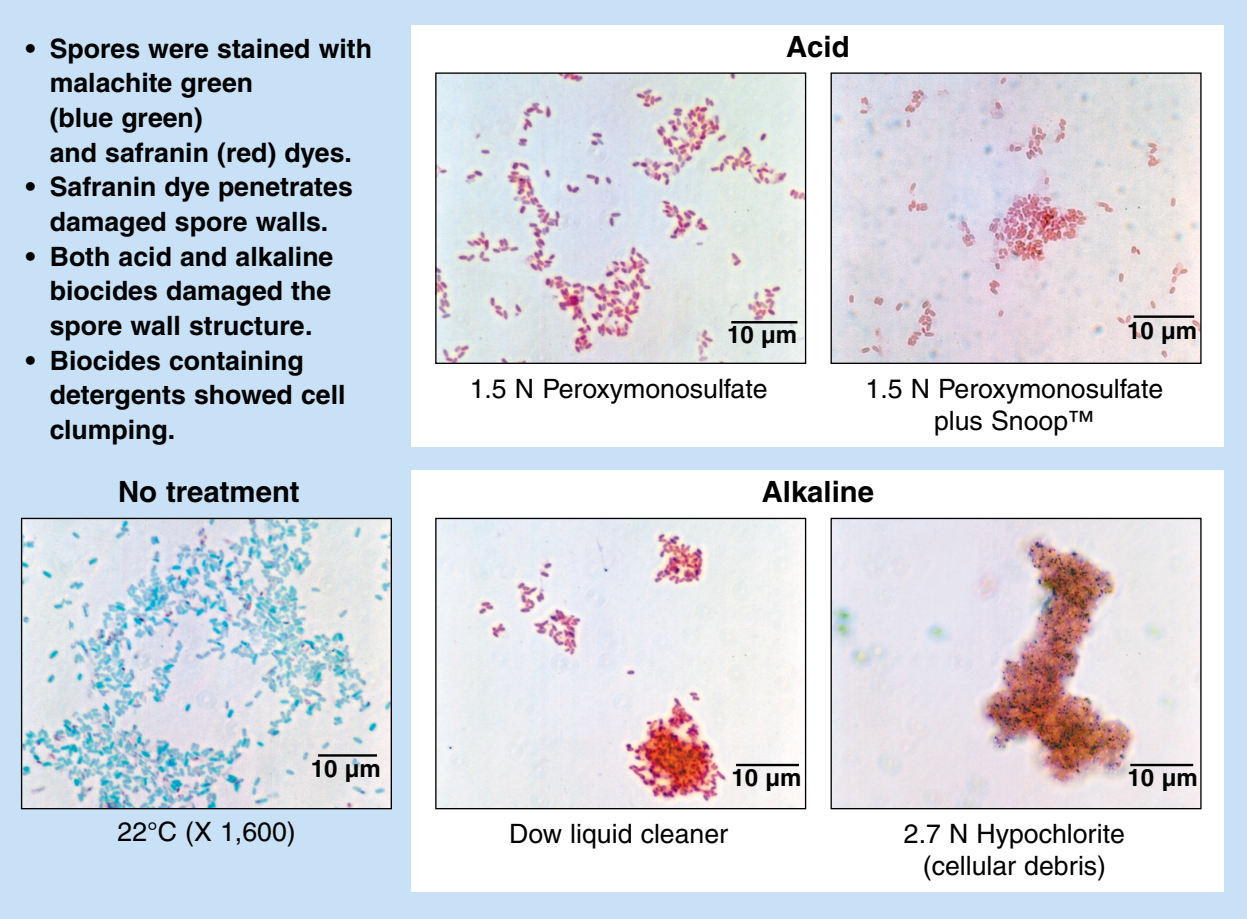

Figure 5. Effect of acid and alkaline oxidizers on BG spore wall structure.

In addition to these tests on BW surrogates, Appendix C shows the results of gel electrophoresis following oxidation/deactivation of ovalbumin (the biological toxin simulant) by 0.01 N Oxone.

The initial laboratory tests of oxidants, together with environmental and practical considerations, eventually led to our selection of Oxone™, whose active ingredient is potassium peroxymonosulfate. This commercial product, which is manufactured by DuPont, has the chemical formulation $2KHSO_5 \bullet KHSO_4 \bullet K_2SO_4$.

2.3.2 Laboratory Testing on Substrate Materials

A series of gelled oxidation experiments using CW and BW surrogates was performed on several test substrates that would be expected in an actual decontamination scenario. Test substrates included glass, fiberglass, varnished wood, acrylic painted metal, and carpet. A surrogate was placed on the test material, and the reagent gel was added to the surface and allowed to dry. All experiments included appropriate laboratory controls and standards. Figure 6 shows the setup for the laboratory experiments to evaluate CW agent surrogates and that used 0.8 N Oxone and amorphous fumed-silica gelling agent.

Figure 7 shows the percent of extracted CW agent, and Figure 8 shows the percent survival of BG spores following decontamination and assessment of the test substrates. These experiments showed that peroxymonosulfate oxidizer gel is 100% effective for decontamination of the CW and BW agent surrogates tested (the only exception was 95% effectiveness against Amiton on carpet).

Figure 6. Lab setup for gelled oxidation experiments on test substrates.

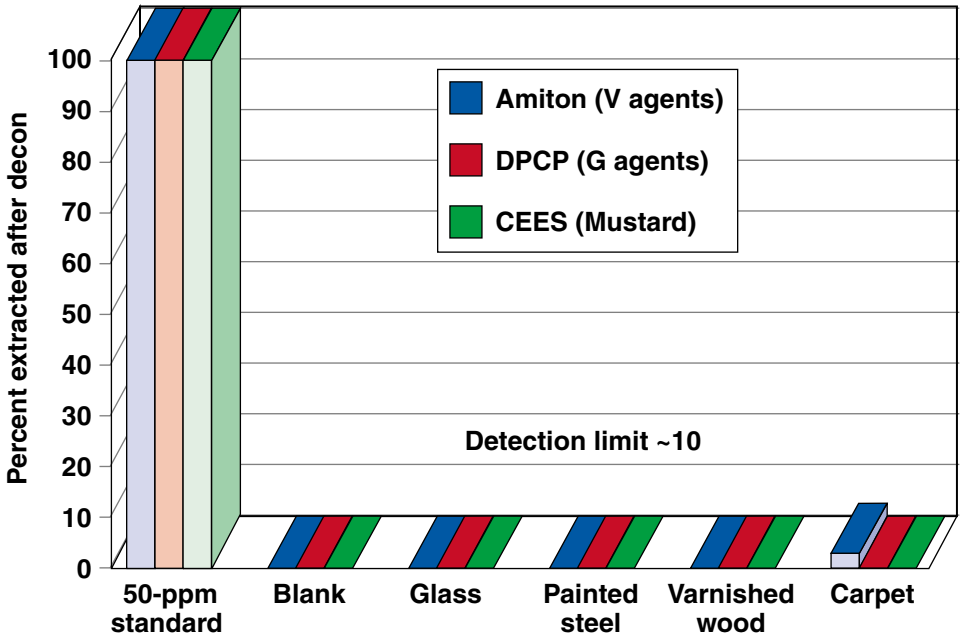

Figure 7. Percent of extracted CW agent from substrates after decontamination.

The biocidal effect of peroxymonosulfate in EH-5 gel on BG spores is illustrated in Figure 9. This image shows a nutrient agar pour-plate of BG spores with three gel spots. Two of the spots contained peroxymonosulfate and one did not. The peroxymonosulfate-containing gel inhibited spore germination in the zone surrounding the gel, indicating leaching of the biocide into the agar.

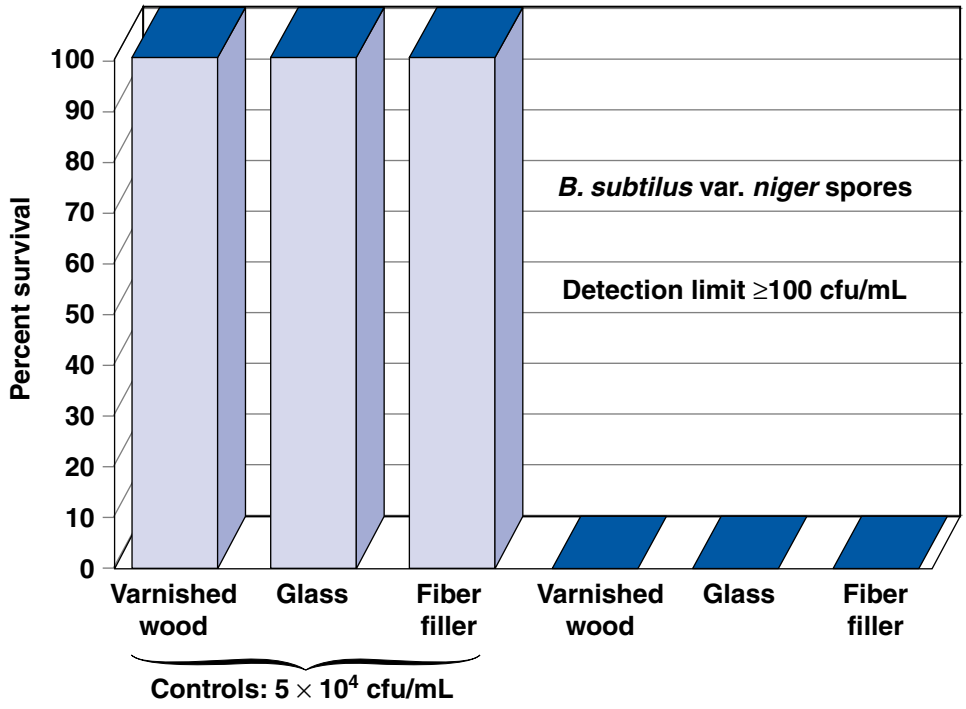

Figure 8. Percent survival of BG spores from substrates after decontamination.

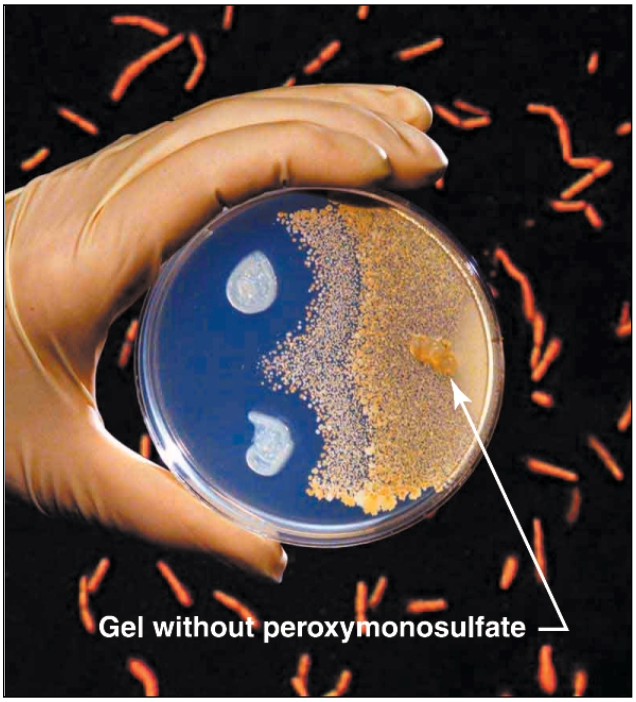

Figure 9. Biocidal effect of peroxymonosulfate on BG spores.

2.3.3 Laboratory Testing of Nonvirulent Strains of Real BW Agents

LLNL obtained nonvirulent strains of live *Bacillus anthracis* spores (Sterne) and *Yersinia pestis* (strain D27) for real-agent testing of decontaminating agents. These anthrax and plague agents were verified by polymerase chain reaction (PCR) identification techniques. LLNL then tested peroxymonosulfate against the strains in a series of lab tests conducted during mid-2000.

Tests were performed by adding ~10^8 cells (or spores in the case of *B. anthracis*) into liquid agar, then pouring onto a Petri plate containing the nutrients required for cell growth. The strains were tested against dilutions of peroxymonosulfate for 30 min, Virkon S, or water. If no decontaminating agent (i.e., only water) is added to the plate, confluent growth is seen, represented by a turbid or opaque agar. In contrast, a zone of inhibition (a clear zone) is created around the disk containing biocidal agent. The diameter of the zone of inhibition is an indication of the sensitivity of the strains to the biocidal agent being tested.

As shown in Figure 10, resistance testing with BG spores and *Y. pestis* suggested that *Y. pestis* is somewhat more difficult to kill with Oxone gel than with commercially available Virkon S. In each case, a 1 N concentration of biocidal agent produced an area of inhibition larger than that for either a 0.5 or 0.1 N concentration. As shown in Figure 11, resistance testing with *B. anthracis*

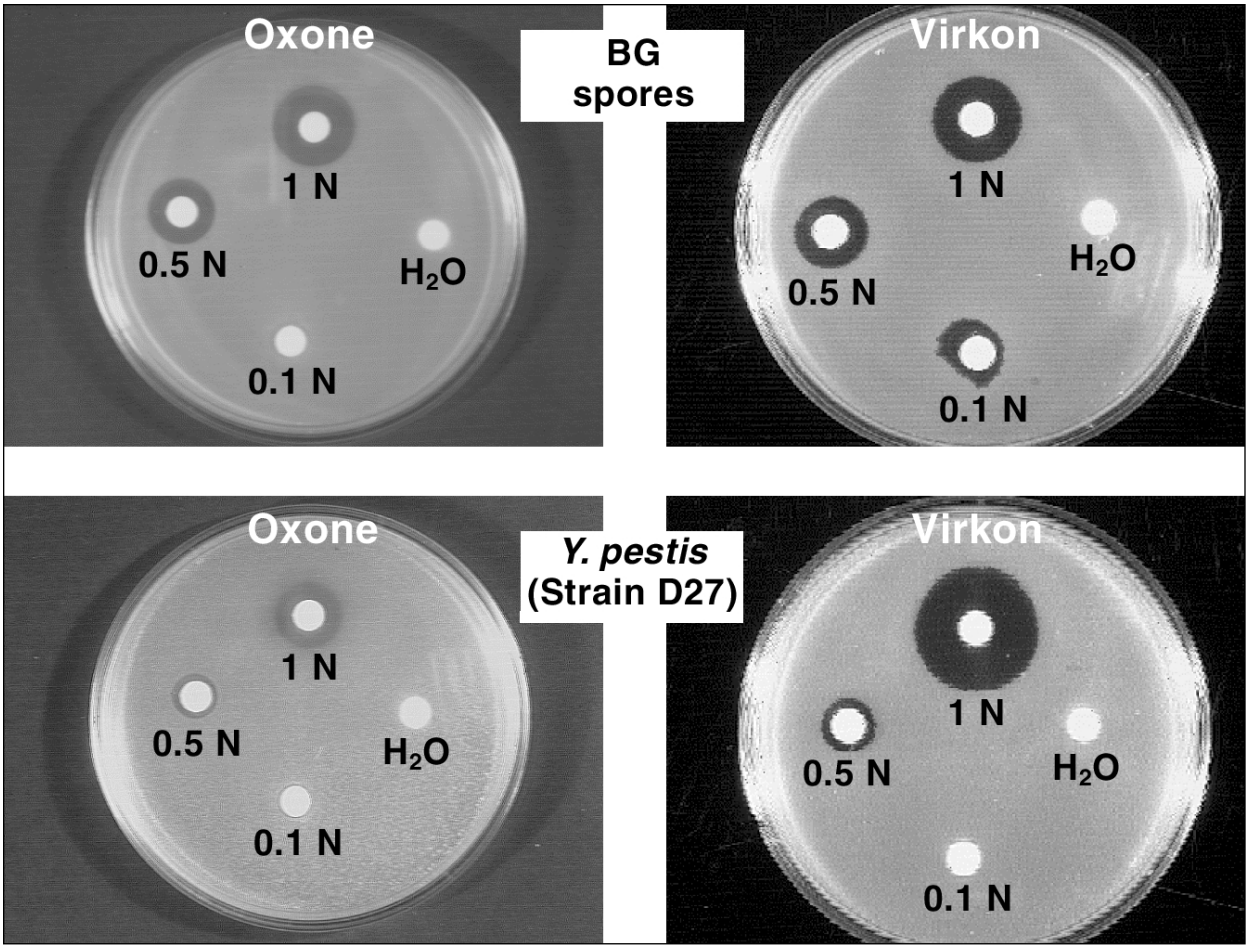

Figure 10. Resistance testing with BG and *Y. pestis*.

spores showed that peroxymonosulfate does effectively kill this BW agent. Standard quantitative methods also revealed a significant difference in spore destruction, namely, that *B. anthracis* is more resistant to decontaminating agents than is BG.

The agar plate resistance test is a standard technique to measure the efficacy of antibiotics. In addition, other experiments (e.g., Figure 4) had previously demonstrated the killing of BG spores by potassium peroxymonosulfate. Nonetheless, it should be noted that Figures 9–11 alone do not definitively show the killing of BW agents by either Virkon solution or Oxone gel, and that the precise mechanism of growth inhibition during the plate tests is not known for either our biocidal agent or for Virkon without performing additional experiments. Control experiments would be necessary to rule out the possibility that these reagents, which are oxidizers, are reacting with components of the plate growth medium and depleting nutrients required for growth.

2.4 Catalytic Nature of Fumed Silica Surface

The hydrolysis of G agents proceeds very rapidly in basic media but is very slow in acidic media, as previously discussed. During experiments with the G surrogate, DPCP, we observed that whereas the decomposition proceeded slowly in aqueous Oxone, it went quite rapidly in the gel. This observation led us to believe that the surface of the gelling acts as a catalyst for hydrolysis. We tested this hypothesis by preparing a series of suspensions of fumed silicas (5% by weight) in aqueous Oxone. The suspensions were allowed to react with DPCP for three hours. The reaction mixtures were then extracted with methylene chloride and analyzed by gas chromatography and mass spectrometry (GC–MS).

Figure 12 shows the residual amount of DPCP (area under the GC peak) as a function of surface area of the fumed silica gelling agent. (For COK-85, only the surface area contributed by the silica was counted.) The linear dependence of the rate of hydrolysis of DPCP in the gelled Oxone with surface area of the gelling agent demonstrates the catalytic nature of the surface. Details of the mechanism of catalysis have yet to be determined.

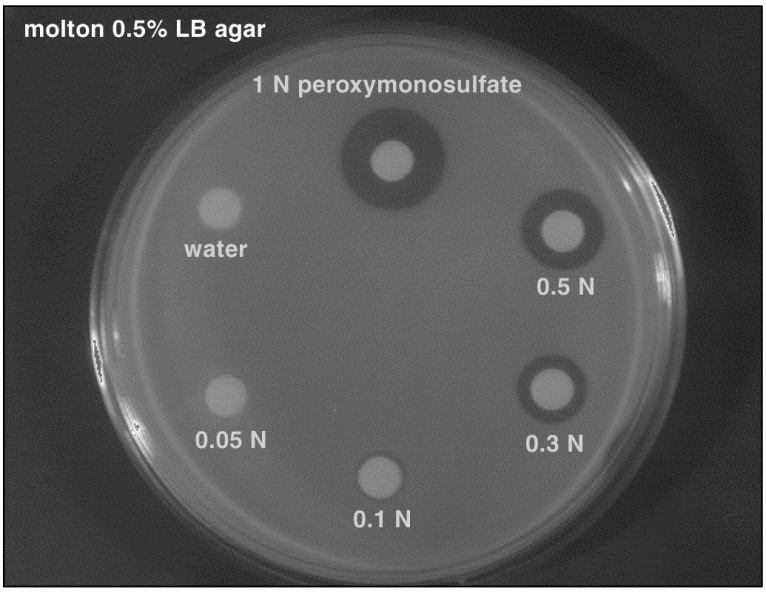

Figure 11. Resistance testing with avirulent *B. anthracis* spores.

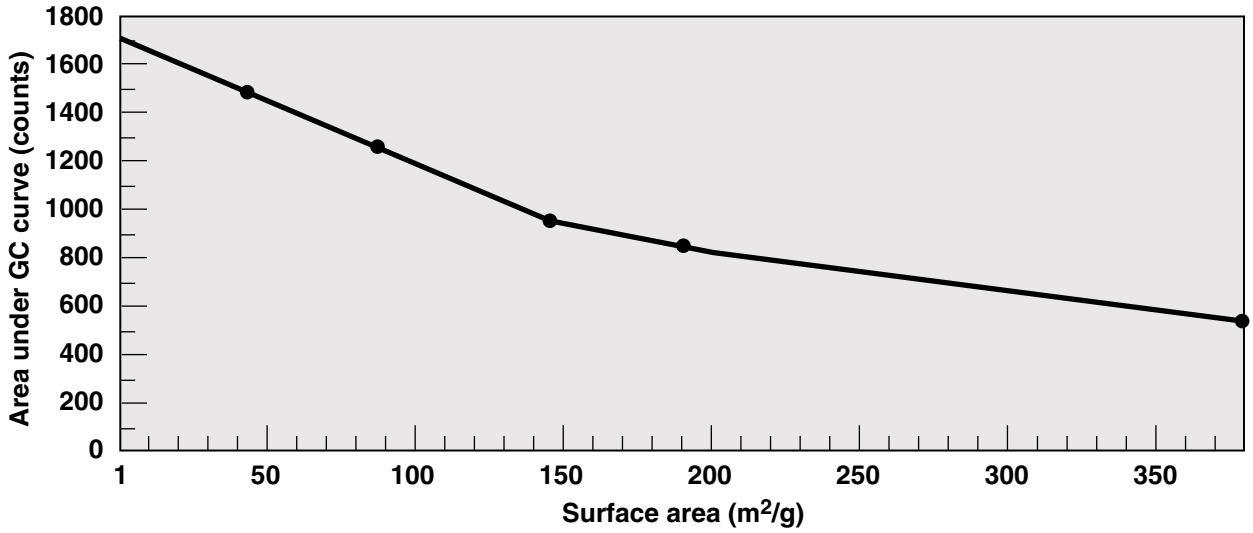

Figure 12. Amount of DCPC after 3 hr in Oxone gel as a function of surface area of gelling agent.

2.5 Application Systems

The successfully developed gel formulation is named "L-Gel." L-Gel-115, for example, is a formulation of 1.0-N aqueous Oxone solution gelled with 15% EH-5. The original concept for dispersal of L-Gel was that it be sprayed as a thickened gel from a high-pressure sprayer. Although a viable high-pressure spray unit was designed, the difficulty of operating such a device in the field suggested that another approach was warranted. Because L-Gel can be easily liquefied by mechanical shaking, we attempted to spray the liquefied gel using a commercial paint sprayer. Both airless and compressed air sprayers were successfully employed.

Our current concept of operation, shown in Figure 13, begins with a storage container of premixed L-Gel in the form of a convenient and transportable solid that does not spill. The container is equipped with a mechanical stirrer so that the gel can be liquefied immediately before use. The gel can then be sprayed using essentially any type of commercial spray device with just about any type of atomizing nozzle. For example, an airless Wagner Power Painter (hand-held model) has been used for smaller applications, or a Graco Electric Airless Paint Sprayer (Model XR7 on wheels) or equivalent has been used for larger applications (~ 5 gal). Stainless-steel nozzles are

Figure 13. L-Gel is shipped premixed as a solid and can be liquefied in 2 to 3 minutes.

recommended. Figure 14 shows the spray pattern achieved with a hand-held Wagner Power Painter. The concept of operation for larger applications, shown in Figure 15, has been demonstrated in field trials. During the development stage of testing an airless Power Painter, we used water formulations containing from 12.8 to 25% EH-5 and sprayed within one day of formulation. Preliminary work showed that a 12.8% solution of EH-5 gave the thickest coverage, but more work needs to be done to optimize spraying coverage.

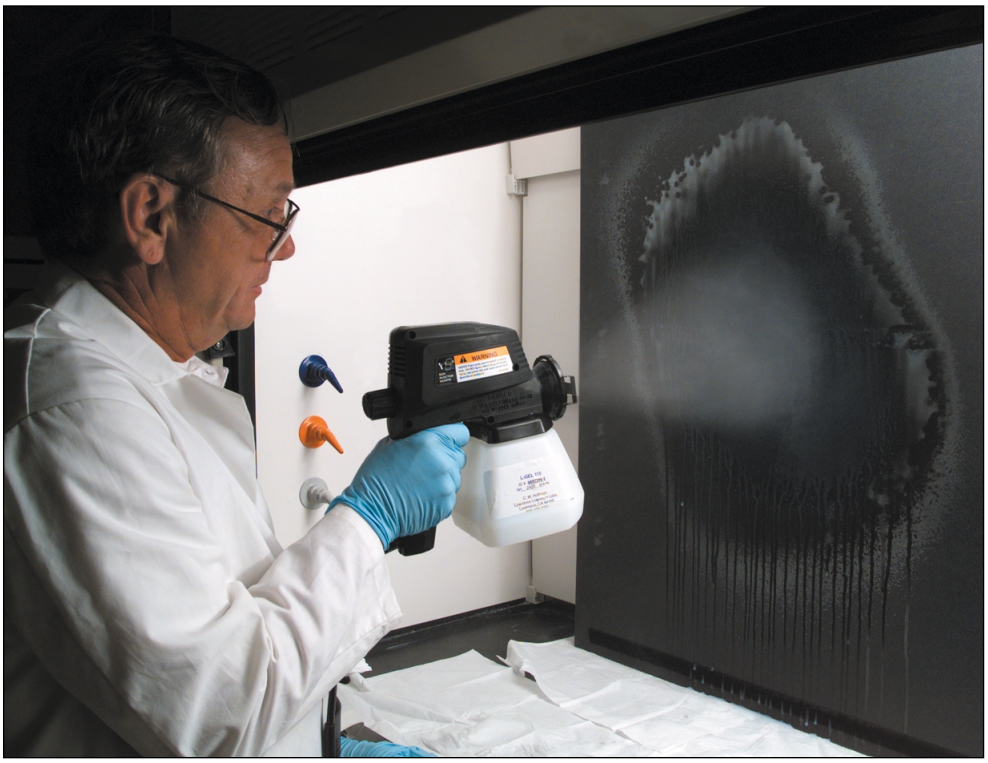

Figure 14. L-Gel spray pattern on a test panel achieved with a hand-held Wagner Power Painter.

Figure 15. Commercially available equipment for L-Gel application. Shown here is a demonstration using a Graco Airless Electric Paint Sprayer.

2.6 Estimates of L-Gel Shelf Life

To assess the shelf life of L-Gel, historical samples that had been used in the field or for other developmental work over the past 2.5 years were analyzed for Oxone content. Both water-based L-Gel-115 samples and mixed-solvent L-Gel-200 samples [~0.5 N Oxone, 15% EH-5, plus tertiary butanol (*t*-butanol) as co-solvent] were analyzed. The L-Gel-200 formulation was specifically designed for decontamination of thickened CW agents, as discussed later in this report. Figure 16 shows the concentration of Oxone as a function of time since the date of formulation.

A simple linear regression fit the L-Gel-115 data reasonably well. (Data are insufficient in the 2- to 3-month regime to determine whether or not the linear fit is accurate over such short times.) The resulting equation for L-Gel-115 was $N = 1 - 0.258(t)$, where N is the normality of Oxone in the L-Gel, and t is time in years. From this equation, it is clear that the normality of Oxone in L-Gel-115 falls at a rate of about 25% per year (assuming linearity) when stored indoors at ambient conditions. After two years, the normality of L-Gel-115 is reduced from 1.0 to 0.5.

Based on the limited data shown in Figure 16, the shelf life of the co-solvent L-Gel-200 system is substantially shorter than that of L-Gel-115, and is probably limited to only 1 to 2 weeks. If the L-Gel-200 system were fielded to defeat a thickened agent, the co-solvent should be added immediately prior to use. However, because of the rapid decomposition rate of L-Gel-200, other organic co-solvents should be investigated, including those that may be more oxidation resistant than *t*-butanol, for example, a water miscible fluorocarbon. Alternate solvents might allow more Oxone to remain in solution, increasing the concentration of active ingredient delivered to a CW or BW agent.

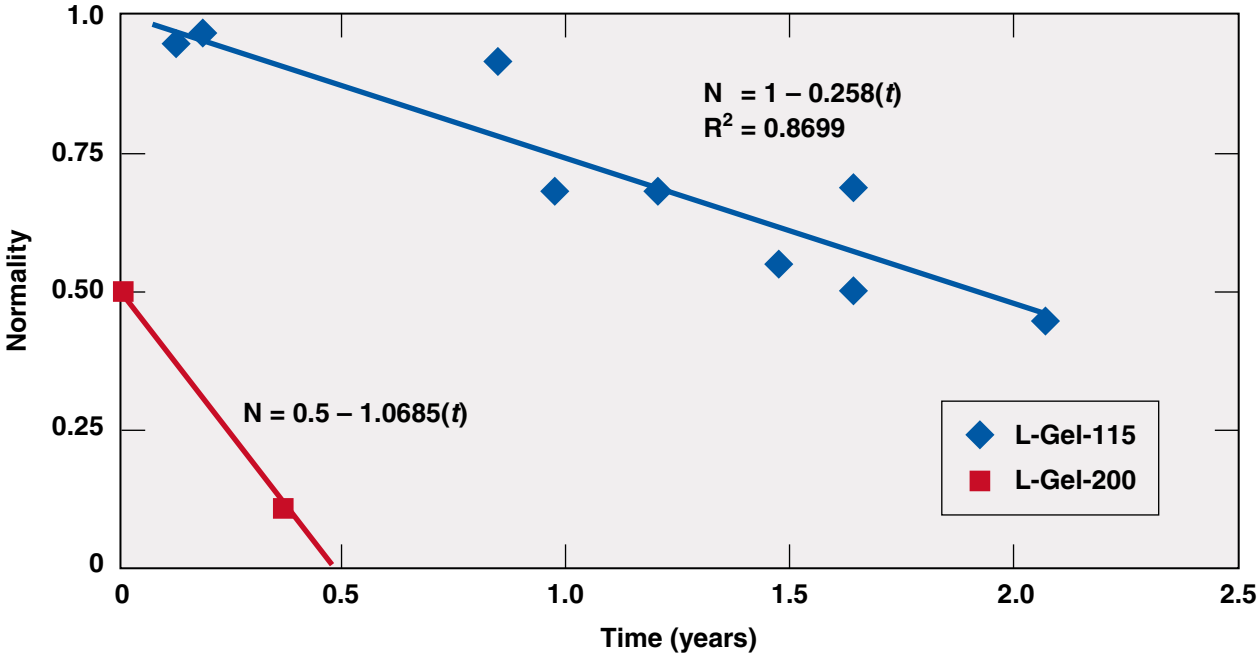

Figure 16. Change in Oxone normality as a function of time since formulation.

3. Field and Laboratory Testing of L-Gel Against Real CW Agents

Given the demonstrated effectiveness of L-Gel against surrogates for all classes of chemical agents, we then tested the effectiveness of L-Gel against real chemical agents. Chemical agent testing was performed independently at four locations:

- Field testing at the Military Institute of Protection, Brno, the Czech Republic (October 1998).
- Lab testing at Edgewood Chemical Biological Forensic Analytical Center (ECBC), Aberdeen Proving Ground, MD (November 1999).
- Lab testing with thickened agents at the Defense Evaluation and Research Agency (DERA), Porton Down, UK (October 1999).
- Field testing with thickened agents at U.S. Army Dugway Proving Ground, UT (July to October 2000).

3.1 Chemical Agent Testing in the Czech Republic

The initial field tests against real chemical agents were performed at the Military Institute of Protection, Brno, the Czech Republic, as part of a series of outdoor tests to evaluate various agent detectors and decontaminants. Four decontaminants were tested on concrete and asphalt against VX and GD. The decontaminants were a water solution of calcium hypochlorite (HTH, a standard military decontaminant that served as a baseline), a Czech emulsion of the HTH, a foamed formulation of the oxidizer Fichlor, and L-Gel-115.

Tables 3 and 4 summarize results of the experiments on asphalt and concrete, respectively. As can be seen by the data, L-Gel-115 performed as well as, or better than, the current military standard, HTH, with the exception of VX on new asphalt. However, because of the uncertainty of the extraction procedure, Table 3 and 4 data should be interpreted only in relation to other data within a table, not as absolute numbers. Appendix D provides a more detailed description of the experiments and analytical method.

3.2 Chemical Agent Testing at the ECBC Forensic Analytical Center

A more controlled, but smaller-scale, series of experiments was performed at the ECBC Forensic Analytical Center. The studies included (1) reactor tests to determine kinetics and reaction products and (2) panel tests on a variety of surfaces.

Reactions were carried out in vessels maintained at 25°C and fitted with mechanical stirrers. Typically, 50 mL of L-Gel-115 decontaminant was added, followed by 1 mL of chemical agent (VX, GD or sulfur mustard HD). Table 5 summarizes the data for 10- and 60-minute reaction times before the reactions were quenched. L-Gel-115 was highly effective against agents VX and HD, but not as effective with GD.

Table 3. Decontamination experiments of real agents VX and GD on asphalt.

New asphalt	VX (% destroyed)[a]	GD (% destroyed)
Baseline [5% $Ca(OCl)_2$]	72	80
HTH emulsion	77	99.8
Foamed Fichlor	56	97
LLNL L-Gel-115	69	98
Old asphalt		
Baseline [5% $Ca(OCl)_2$]	–	95
HTH emulsion	–	99
Foamed Fichlor	–	98
LLNL L-Gel-115	–	98

[a] VX was only tested on new materials.

Table 4. Decontamination experiments of real agents VX and GD on concrete.

New concrete	VX (% destroyed)[a]	GD (% destroyed)
Baseline [5% $Ca(OCl)_2$]	95	100
HTH emulsion	98	100
Foamed Fichlor	83	100
LLNL L-Gel-115	99	100
Old concrete		
Baseline [5% $Ca(OCl)_2$]	–	95
HTH emulsion	–	99
Foamed Fichlor	–	97
LLNL L-Gel-115	–	98

[a] VX was only tested on new materials.

Table 5. Percent reaction of VX, GD, and HD after 10 and 60 minutes in ECBC reactor tests.

Time	VX (% reaction)	GD (% reaction)	HD (% reaction)
10 minutes	100	20	>99
60 minutes	100	55	100

The agents VX, GD, and HD were investigated on small samples of acrylic painted metal, polyurethane varnished oak, and indoor-outdoor carpet. In all cases, L-Gel-115 was allowed to dry before samples were extracted with methylene chloride and analyzed by GC–MS.

The results showed that the agents were completely destroyed, except for GD on both of the painted surfaces. For the polyurethane surface, 6% of the GD was recovered; for the acrylic surface, 20% of the GD was recovered. This result is not unexpected and shows the effect of solubility of GD in paint. The polyurethane, a harder surface, dissolves less GD, allowing more of it to be destroyed.

A further set of experiments was performed with GD on painted surfaces, where a small amount of methylethyl ketone (MEK) was added to the L-Gel just before use. The amount of recovered GD was markedly reduced despite the fact that the decontaminating reagent was more than 5 months old. The GD from the polyurethane surface was reduced to 4.3% and from the acrylic surface to 11.4%. A comparison of the results on painted metal with those on unpainted metal (2.42% GD recovered) indicates that the problem is the solubility of GD in the paint. Details of these experiments are given in Appendix E.

3.3 Experiments with Thickened Agents at Porton Down

A series of experiments was performed against thickened GD (TGD) and thickened Mustard (THD) at the Chemical and Biological Defense Establishment (CBDE), Defense Evaluation and Research Agency (DERA), Porton Down, UK. Thickened agents were applied to metal plates measuring approximately 3 × 5 in. and painted with either an alkyd paint or a polyurethane paint. The agents were allowed to remain in contact with the surfaces for one hour before attempting decontamination. Following this time, L-Gel-115 was sprayed on the samples in the vertical position, using a commercial British compressed air paint sprayer. A contact time of 30 minutes was allowed before the sample panels were sprayed with ambient-temperature water at high pressure. The panels were then placed in a measured amount of isopropanol for two hours, and the extract was analyzed by GC–MS.

Table 6 summarizes the results, which show the effect of decreased diffusion from a combination of agent thickener and agent solubility in the painted surfaces. Given the shorter reaction times and the use of thickened agents, the results are consistent with previous results from tests on painted surfaces obtained by ECBC Forensic Analytical Center.

Table 6. Results of thickened agent decontamination tests with L-Gel-115.

Thickened agent	Alkyd paint	Polyurethane paint
TGD	35% destroyed	64% destroyed
THD	50% destroyed	66% destroyed

3.4 Experiments with Thickened Agents at Dugway Proving Ground

We developed a second formulation (L-Gel-200), which contains a co-solvent to promote solution and oxidation for thickened CW agents. In mid- to late 2000, the L-Gel-200 formulation was field tested against real CW agents as part of the Restoration of Operations (RestOps) series of experiments conducted at the West Desert Test Center, U.S. Army Dugway Proving Ground in Utah. LLNL sent L-Gel-200 materials, associated delivery equipment, and participated in personnel training exercises.

The initial RestOps experiments evaluated various technologies for their ability to decontaminate 16-gauge mild steel test panels painted with Army/Marine chemical agent resistant coating (CARC) (Mil-C-53039), Air Force air ground equipment (AGE) paint (Mil-PFR-85285), and Navy shipboard coating (Mil-E-24635). The panels were contaminated with thickened distilled mustard (THD), thickened soman (TGD), or persistent nerve agent (VX).

Because of the difficulties of water-based decontamination against thickened agents, L-Gel-115 was modified by the addition of 10% co-solvent (*t*-butanol), which is known to be resistant to oxidation compared to other organic solvents. Previous evaluation of L-Gel-115 against THD and TGD in the UK had shown decreased diffusion and reduced detoxification compared to neat GD or HD. Similar results were obtained at ECBC with GD on acrylic paint. When MEK was added just prior to spraying L-Gel, a factor of 2 improvement in detoxification of GD on the acrylic surface was observed. Because an acrylic copolymer is used to thicken chemical agents, this approach seemed promising. Rapid oxidation of MEK would require mixing immediately before application, so L-Gel-200 was prepared with *t*-butanol instead. The addition of a co-solvent also reduces the

solubility of Oxone in the solution to 0.5 N. The L-Gel-200 formulation (0.5-N Oxone with 15% EH-5 fumed silica) was formulated and scaled up to 5 gallons for shipment to Dugway Proving Ground.

An attempt was made to premix the EH-5 and water several months prior to adding the Oxone and *t*-butanol. High-bulk-density fumed silica (EH-5) can be prepared and stored easily as a 17.4% water gel. We originally believed that this gel could be kept for long periods to extend the shelf life of L-Gel-115. If Oxone degradation were the only issue, water gels would work. Unfortunately, the gelation of EH-5 in water over time caused it to loose its thickening ability. This may be due to loss of silanol groups on the surface of the silica, which react irreversibly during gelation. Because of the differences observed in the premixed formulation of L-Gel-200, a 1 gallon mix was made from scratch and sent to Dugway for evaluation. The original 5 gallon premix formulation was used to train personnel and evaluate the sprayer system at Dugway, and the fresh mix was used for decontamination experiments.

The Dugway panel tests used 4.4-cm-diameter carbon steel panels painted with the appropriate coating and mounted in a holder. Panels were contaminated with a specific agent using a syringe with at least 10 g/m^2 contamination density (concentration of the agent was approximately 10 mg). The agent was streaked onto the panels rather than applied as droplets. Additional coated panels with no agent applied served as negative controls. Each panel was covered after contamination; once all panels had been contaminated the covers were removed, and the agent was allowed to weather for 1 hour.

Contaminated panels were sprayed with L-Gel-200 using a Grayco Magnum XR-7 high-pressure paint sprayer with extender and RST 15 tip until they were completely covered. Panels were allowed to dry for 30 minutes and resprayed two more times in the same manner. Each panel was removed and placed in a jar with 10 mL of methylene chloride extraction solvent. Samples were analyzed by GC–MS for the agent of interest. Runoff from the test was collected and extracted with 2 mL of chloroform and analyzed by GC–MS for the agent of interest.

Table 7 and Figure 17 show the co-solvent L-Gel-200 results from Dugway RestOps panel tests against thickened or persistent agents. Of the three coatings tested, the Navy shipboard paint was the most difficult to decontaminate. Although the normality of co-solvent-based L-Gel-200 is only half that of L-Gel-115, these results represent about a factor of 2 improvement compared to L-Gel-115 data on thickened agents from Porton Downs.

Table 7. Panel test results for L-Gel-200 co-solvent system against THD, TGD, and persistent VX chemical agents.

	Percent destroyed		
Paint	**THD**	**TGD**	**VX**
CARC	82.3	91.8	90.9
Navy shipboard	64.4	75.3	80.3
AF ground equipment	68.6	95.5	90.9

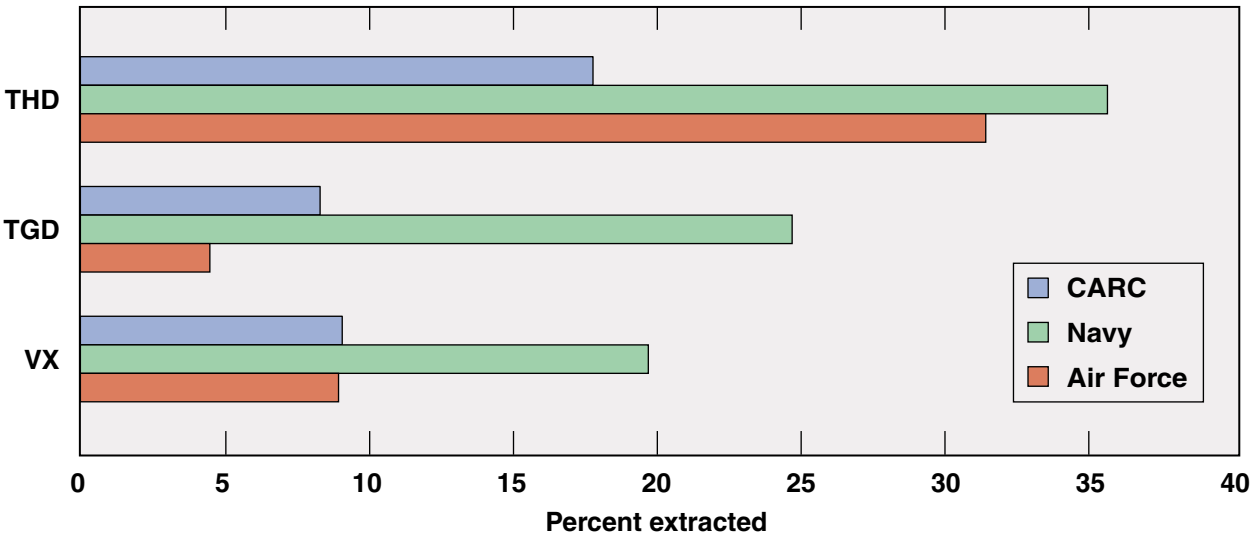

Figure 17. Dugway co-solvent L-Gel-200 results against thickened and persistent chemical agents.

4. Field Testing of L-Gel Against BW Surrogates

Following the laboratory-demonstrated effectiveness of L-Gel against surrogate bacterial spores, we field tested the effectiveness of L-Gel against BG spores in two separate field exercises.

4.1 Surrogate BW Agent Panel Testing at Dugway Proving Ground

In December 1999, LLNL participated in BW field tests conducted by the Soldiers Biological and Chemical Command at the U.S. Army Dugway Proving Ground, West Desert Test Center, UT. The test objectives (Larson and Harper, 1999) were to compare the ability of several candidate decontamination materials to inactivate a BW agent simulant. In all, seven candidate decontaminating technologies from research facilities around the country were evaluated. The five days of testing were the first time that a direct comparison of the seven technologies was made.

A major issue is to understand what level of biological decontamination is achievable in a civilian office setting, and what level is acceptable. As far as criteria are concerned, the military scenario (U.S. Army Regulation 70-75) requires that an initial contamination density must be reduced by at least 200-fold, with no more than 500 spores/m^2 remaining.

The six selected materials consisted of 16- by 16-in. panels that were contaminated with ~$1 \times 10^9/m^2$ of BG spores each evening and sampled by standard swab testing each morning. Between 11 and 12 a.m., L-Gel was applied, and about 24 hours later, the panels were reswabbed,

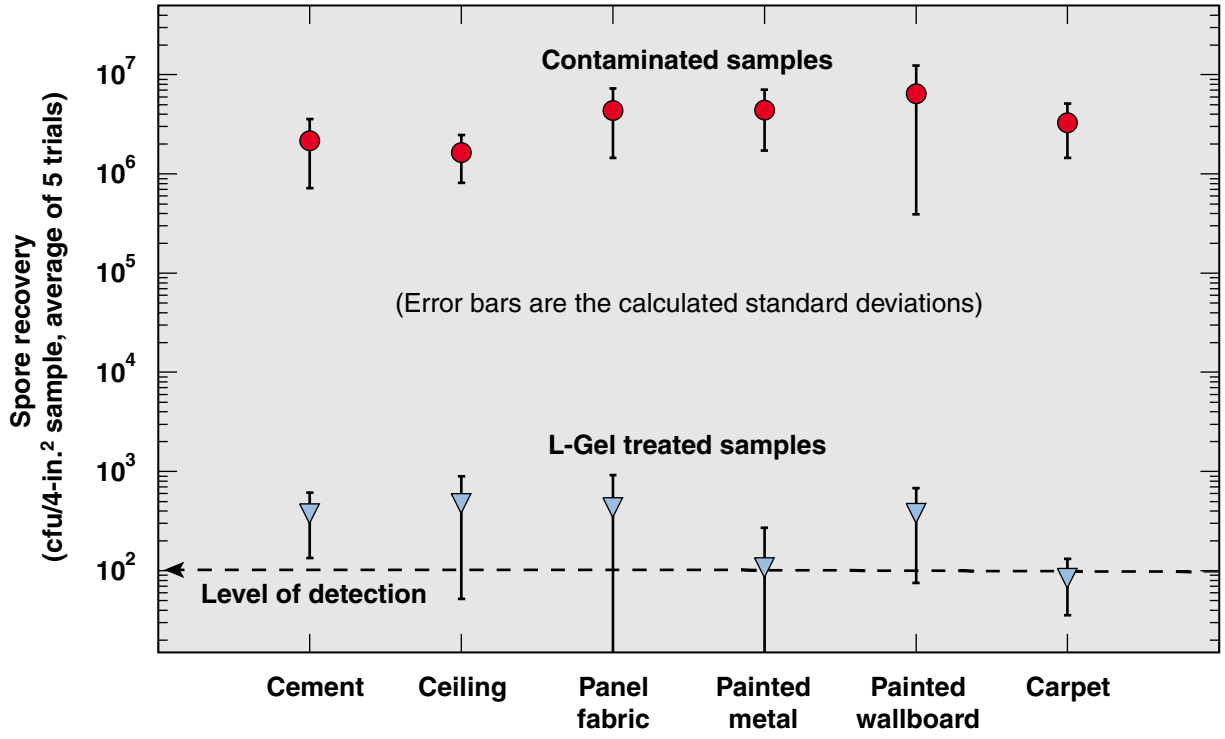

Figure 18. Results of Dugway field tests on six materials contaminated with BG spores before and after application of L-Gel.

and the process was repeated. The six panels included acoustic ceiling tile, tightly woven carpet, fabric-covered office partition panel, painted wallboard, concrete block slab, and painted metal. Tests were repeated five times in one practice test and four replicate tests. Details on the test methodologies are given in Larson and Harper (1999) and Appendix F. As shown in Figure 18, L-Gel performed very well in these Phase I panel tests. The number of live spores on test panels was reduced by an average of 99.988% through the application of L-Gel-115.

4.2 Surrogate BW Agent Mock Office Decontamination at Dugway Proving Ground

In October 2000, LLNL participated in the BW agent room-decontamination exercise coordinated by the Soldiers Biological and Chemical Command at the U.S. Army Dugway Proving Ground, West Desert Test Center, UT. On the basis of test results from the December 1999 panel tests, three organizations were selected for testing of their decontamination methods on full-scale mock office spaces, as shown in Figure 19.

Each of six 8-ft-square mock offices was built in an abandoned building. Flooring was divided into quarters consisting of carpet, vinyl tile, oak flooring, and painted concrete. Walls consisted of stucco, wood paneling, sheet rock, and carpet. The ceiling was constructed of suspended ceiling tile. The room was contaminated with 4 g of BG spores by a simulated explosion using a disseminator, and spores were distributed by an oscillating fan.

Approximately 400 samples were collected by swabs from multiple locations, one sample per square foot. Swabs were placed in sterile, buffered solution containing sodium thiosulfate. The diluent was plated onto TSA agar, and live colonies were counted. The detection limit of the analyses was 1×10^2 colony forming units (cfu) per 4 square inches.

Figure 19. Mock office used for evaluation of L-Gel at Dugway, UT.

Figure 20(a) shows that the distribution of BG on the floor of the first contaminated mock office was fairly uniform. More spores were found on the floor than on any other surface. The distribution of BG on the floor of the mock office used to evaluate L-Gel had about the same mean value as that of the original mock office [compare Figure 20(a) and 20(c)]. L-Gel's performance was comparable to that of the paraformaldehyde treatment in eliminating BG from the mock offices [compare Figure 20(b) with 20(d)]. Paraformaldehyde reduced the distribution of BG on the floor by about 5 orders of magnitude, as did decontamination with L-Gel-115. The L-Gel did not bleach or damage office surfaces, with the exception of some rust on ceiling supports.

During the testing of decontaminating reagents against BG spores at Dugway, we noted a possible source of bias in the method used to recover spores for evaluation. The quenching agent, sodium thiosulfate, contained in the buffered solution affects oxidizers, such as peroxymonosulfate in L-Gel. The two other technologies tested in the exercise contained decontaminating agents (lipid disrupters and enzymes) that are not quenched. Thus, it is possible that the side-by-side comparison of decontaminating agents was flawed by sodium thiosulfate. This issue needs to be investigated further.

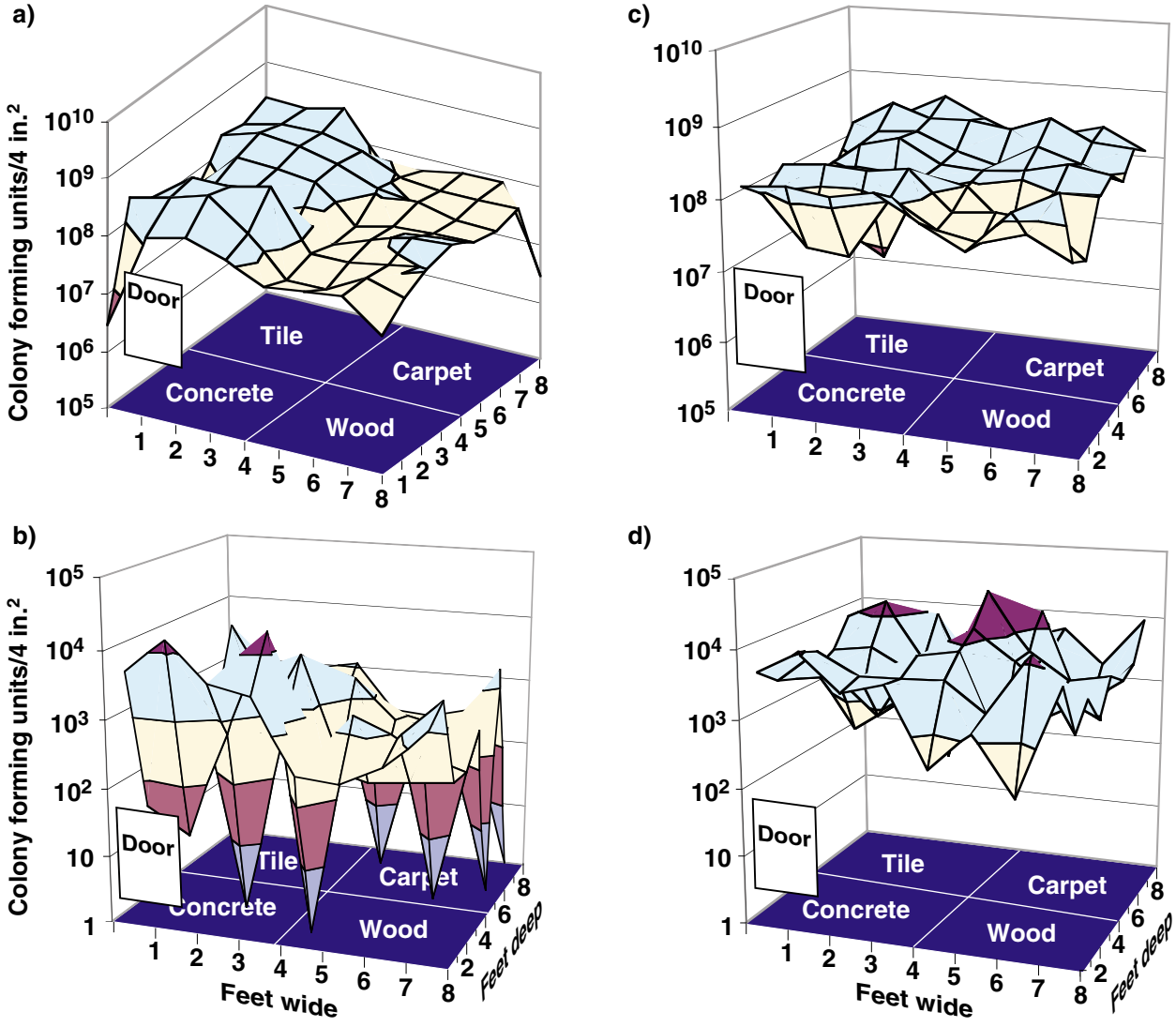

Figure 20. Distribution of BG on the floor of mock offices (a) before and (b) after decontamination with paraformaldehyde, and (c) before and (d) after decontamination with L-Gel-115.

5. Summary

Experimental studies to date show that the L-Gel system developed by LLNL is effective for both CW and BW decontamination.

- Seven oxidizers were lab tested for decontamination against CW surrogates, Amiton, DPCP, and CEES, in concentration- and time-dependent studies. The L-Gel system was 100% effective on all tested substrate materials (varnished wood, painted steel, glass, and carpet) and 95% effective for Amiton on carpet.

- Field tests using real CW agents VX and GD show that L-Gel is more effective against those agents than a standard U.S. military method (using HTH).

- Field tests using VX, GD, and Sulfur mustard (HD) on small samples of four materials showed that the agents were completely destroyed, except for GD on the two painted surfaces. The problem is the solubility of GD in paint.

- An L-Gel-200 formulation containing 10% *t*-butanol co-solvent to promote solution and oxidation for thickened chemical agents was field tested. Although the normality of co-solvent-based L-Gel-200 is only half that of L-Gel-115, the results showed about a factor of 2 improvement compared to L-Gel-115 data on thickened agents.

- Nine oxidizers were lab tested for biocidal activity against *B. subtilus* var. *niger* (BG) spores in concentration-dependent studies. The three most effective are the peroxymonosulfate system (L-Gel), modified Fenton reagent, and Virkon S. L-Gel was 100% effective without quenching in laboratory tests against the spores on various surfaces (varnished wood, glass, and fiber).

- Ovalbumin (toxin simulant) was oxidized/deactivated in lab tests with low concentrations (0.01 N) of peroxymonosulfate.

- In field tests, live BG spores on test panels were reduced by an average of 99.988% through the application of L-Gel.

- In room-decontamination field tests, L-Gel's performance was comparable to that of the paraformaldehyde standard in eliminating BG from the mock office.

The principal characteristics and advantages of the L-Gel system are as follows:

- L-Gel is effective against all chemical and biological agents.

- The fumed silica gel is compatible with strong oxidizing agents.

- The system is relatively noncorrosive, with a pH approximately equal to that of vinegar or lemon juice.

- The L-Gel system is relatively inexpensive and available (~$1.00/$m^2$).

- L-Gel maximizes contact time because of its thixotropic nature. L-Gel clings to walls and ceilings and does not harm carpets or painted surfaces.

- The ability of L-Gel to liquefy when stirred or shaken and to return to the "solidified" state upon standing enhances material handling, application, and contact time.

- Methods of dispersal are easy and can be varied depending on user needs and required viscosity (200 g/m^2 at a thickness of ~5 mil).

- No complicated equipment is required for preparation or application. L-Gel can be sprayed using a commercially available stainless steel sprayer and atomizing nozzle.

- Drying time is 1 to 6 hours; decontamination is faster, 30 to 40 minutes.

- Dried residue indoors can be vacuumed and discarded. Outdoor use requires no cleanup. U.S. EPA methods (8260/8270 for volatiles and semi-volatiles) show residual byproducts to be nonhazardous.

- L-Gel is expected to have a long shelf life (>1 year) if unopened, allowing it to be premixed.

- L-Gel material meets nonhazardous/noncorrosive DOT requirements and is stable during shipping.

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7. Acknowledgments

The authors wish to thank Robert Kirvel and Brett Clark for their help in editing text and preparing the illustrations for this document.

Appendix A: Experiments on Compatibility of Organic Foam with Oxidizing Agents

In view of the presence of oxidizable organic molecules in organic foam, the problem of compatibility with strong oxidizing agents was considered to be significant. Several experiments were performed to determine the extent of incompatibility. An aliquot of a foam solution was reacted with an aliquot of an oxidizer such that the resulting concentrations of foam was 6%, and the oxidizer concentration ranged from 0.01 to 1.0 N. The combined aliquots were stirred for five minutes, and the remaining oxidizer concentrations was then measured. Table A-1 summarizes experimental results.

Table A-1. Experiments to measure compatibility between various oxidizers and foams supplied by Sandia National Laboratories.

Oxidizer	Oxidizer conc. (N)	Initial pH of oxidizer + anionic foam	Initial pH of oxidizer + cationic foam	Percent oxidizer used up by anionic foam in 5 minutes	Percent oxidizer used up by cationic foam in 5 minutes	Notes[a] on cationic foam	Notes[a] on cationic foam
Hypochlorite (basic)	0.01 0.10 0.30	10.0 11.3 11.8	10.6 11.5 12.0	14 10 9	40 6 2	Foaming reduced as oxid. conc. increased	Foaming reduced as oxid. conc. increased
Hypochlorite (acidic)	0.01 0.10 0.30	3.0 3.0 3.0	3.0 3.0 3.0	97 37 23	33 18 26	Solution still shows foaming action	Foaming reduced as oxid. conc. increased
H_2O_2 + 10 ppm Cu^{2+}	0.01 0.10 1.0	3.0 3.0 3.0	3.0 3.0 3.0	94 77 44	39 22 8	Solution still shows foaming action	Solution still shows foaming action
Peroxy-monosulfate + 10 ppm Cu^{2+}	0.01 0.10 1.0	3.0 1.8 1.5	2.8 1.8 1.0	51 27 —	29 6 5	Solution still shows foaming action	Poor precipitation occurred
Peroxy-disulfate + 10 ppm Cu^{2+}	0.01 0.10 0.30 1.0	3.0 3.0 3.0 2.4	3.0 3.0 2.7 2.1	25 4 1 —	27 4 2 0	Solution still shows foaming action	Poor precipitation occurred

[a] Despite significant reaction with oxidizing agents, certain foams continued to exhibit some foaming action after 5-minute exposures.
The effects of longer exposures were not determined.

Appendix B: Development of Gelled Aqueous Oxidizer Formulations

The gelation characteristics of a particular gelling agent were evaluated in stepwise fashion by adding a weighed amount of deionized water to a weighed amount of silica. The mixtures were agitated for 5 minutes on a paint shaker. The resulting suspension was allowed to sit for 5 minutes and classified on a scale from 1 to 5 based on observed quality of the gel, where 1 = watery liquid, 2 = thick liquid, 3 to 4 = gels of increasing integrity, and 5 = extremely viscous or dry gel. The silica content was increased slowly until gelation occurred. Figure B-1 shows the results for each gelling agent evaluated.

In general, viscosity increased gradually until the concentration of gelling agent was sufficient to form a network and the system gelled, weakly at first then rapidly as concentration of gelling agent increased. The fumed silica systems, EH-5 and A-200, were difficult to disperse with a paint shaker as they neared gelling concentrations. The systems tended to aggregate in inhomogeneous "flocs" at around 12 to 13% by weight. Assessment of the degree of gelation of fumed silica/water systems from mixing with a paint shaker was tenuous at best. These systems also "crape harden" with time. Mixtures of 13% or more EH-5 or A-200, which were fluid after shaking when left overnight or for a few hours, set up into very stiff gels. The precipitated silica (T-700) has a slightly larger primary particle size and therefore slightly lower surface area. For this reason, it gels at greater concentrations than fumed silica in water. Remarkably within the limits of these

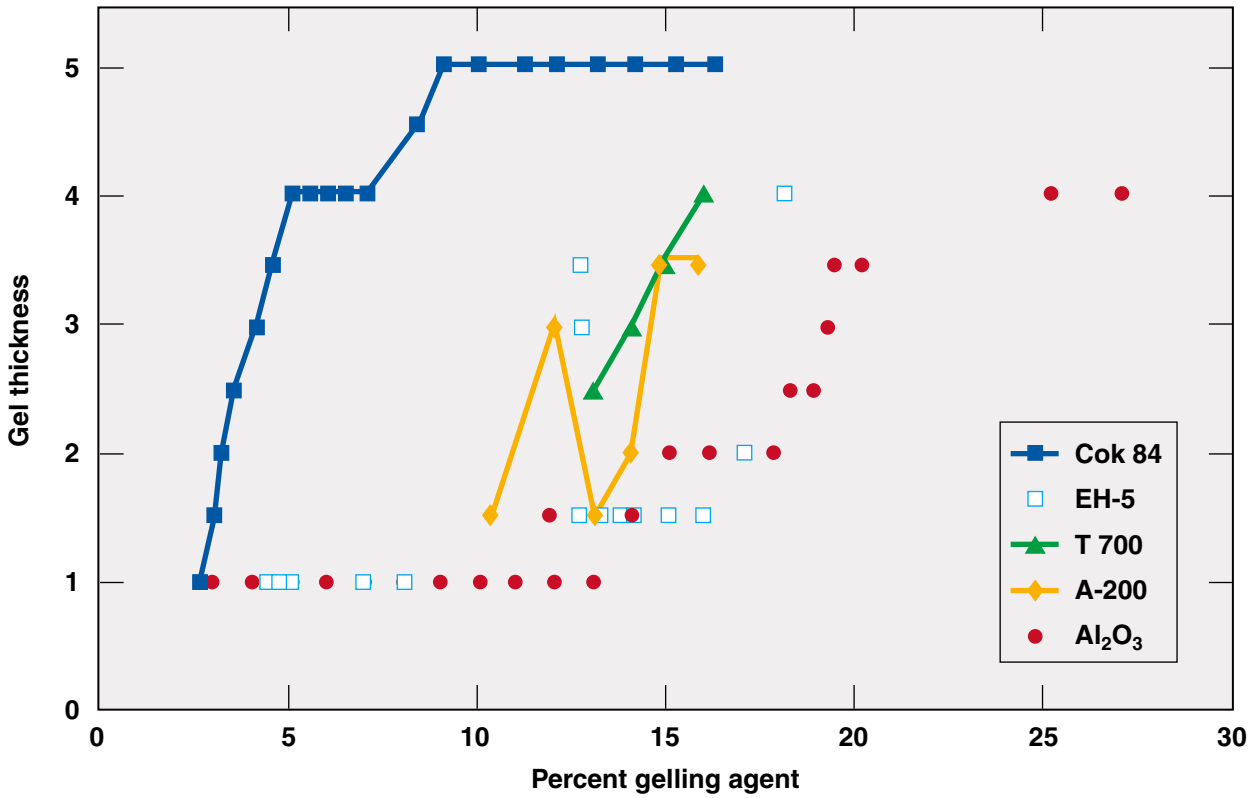

Figure B-1. Classification of various gelling agents.

experiments, the precipitated silica did not show the inhomogeneous flocs or static behavior observed with fumed silica. The COK 84 blend of SiO_2 and Al_2O_3 required the lowest concentration of gelling agent to produce aqueous gels (as low as 4 to 5%). Although the fumed alumina (aluminum oxide C) has a small particle size, it was the least effective gelling agent, indicating the importance of active sites on the colloid. Viable alumina gels required upwards of 25 to 27% by weight gelling agent. Table B-1 summarizes some general characteristics of the various gelling agents that were evaluated.

Table B-1. Gelling agent characteristics based on manufacturer data sheets.

Gelling agent	Particle size (nm)	Aggregate size (µm)	Surface area (m^2/g)	pH
Cab-O-Sil EH-5	7	0.3	380	4
Aerosil 200	12	0.4	200	4
COK 84	-	-	170	4
Alumina C	13	-	100	5
Hi-Sil T-700	21	1.9	210	7

Because of previous experience with EH-5 gelling agent and time limitations, mixed solvent systems were only evaluated with EH-5 fumed silica. Two formulations, 20% solutions of methylethyl ketone and ethyl alcohol in water, were evaluated in the same manner as described above. The curves for degree of gelation as a function of concentration of EH-5 were shifted to higher concentrations for the 20% mixed solvent systems compared to water, but they clearly form suitable gels. The mixed solvent gels were similar. Between 14.8 and 17.8% EH-5, moderately viscous gels formed. These gels were comparable to 12.8 to 14.8% water/EH-5 gels. Although mixed solvents were more readily dispersed by the paint shaker than water based gel, inhomogeneity of floc continued to be a problem. A Silverson high-shear mixer greatly facilitated the formulation of gels.

Several of the oxidizers proposed for decontamination applications (see Table B-2) were evaluated with EH-5 fumed silica as a gelling agent. The gels are similar to those generated without oxidizer, with persulfate and hydrogen peroxide requiring 3 to 4% increase in concentration of gelling agent compared to water. Samples of 13.5% EH-5 gel of 5.5% sodium hypochlorite in water were stored in a hood at ambient temperature and in a refrigerator at 10°C for several months without evidence of gas generation or other degradation. A 14.5% EH-5 gel of 3 N ammonium persulfate was stored in the refrigerator without obvious evidence of degradation. However, when a 16.4% EH-5 gel of 30% hydrogen peroxide was stored under these conditions, bubbles of oxygen were observed within a few days. Similar results occurred with a 5% Cok-84 and 30% hydrogen peroxide gel.

Table B-2. Oxidizers proposed and gelled successfully with EH-5 fumed silica.

Oxidizer	Manufacturer	Percent in solution	Normality	Molecular weight
NaOCl	Clorox	5.5		74.44
NaOCl	Clorox	9.5		74.44
$(NH_4)_2(SO_4)_2$	Aldrich		0.3	228.22
H_2O_2		30	19.2	34.82
Oxone	DuPont			

Appendix C: Selection of Oxidizing Agent

Oxidation of Chemical Agent Simulants

The initial oxidation experiments were carried out using the following general procedure. All oxidizers were made up in deionized water to 0.3 N concentration. The hydrogen peroxide solution was adjusted to a pH of 3 with sulfuric acid. The other oxidants were used at the unadjusted pH of the 0.3 N solution (pH = 3 or less). All oxidizer solutions were stored in amber bottles in a refrigerator until use. A copper sulfate catalyst solution was added immediately prior to a given experiment.

The amount of oxidizer used in each experiment was calculated to provide a 2:1 (oxidizer: surrogate) stoichiometry based on the use of 0.5 µl of surrogate. The oxidizer solutions were added to 15-mL vials using a 10- to 100-µl pipette. The vials were closed with PTFE-silicone septa screw caps.

The reaction time of 30 minutes began with the addition of 0.5 µl of surrogate to the oxidizer solution in the vial. A 0.1- to 2.5-µl pipette was used for surrogate addition. Vials were then recapped and placed on a shaker, running at about 3 cps for the remainder of the 30-min reaction time. The pipette was rinsed several times with oxidizer solution prior to subsequent use.

Reactions were quenched by adding 10 mL of methylene chloride to the vials and shaking an additional 10 minutes to extract any unreacted surrogate. The total volume of methylene chloride was chosen so that the concentration of surrogate to be analyzed would be approximately 50 ppm if no oxidation took place. Samples were then analyzed by GC-MS using a Saturn Ion Trap instrument with a DB-5ms column.

To obtain representative, repeatable data for the materials Amiton and DPCP, it was necessary to inject the standard solution into the GC at least three times to "prime" the column. Figure C-1 shows an example of this phenomenon for Amiton. A series of three to five solvent injections

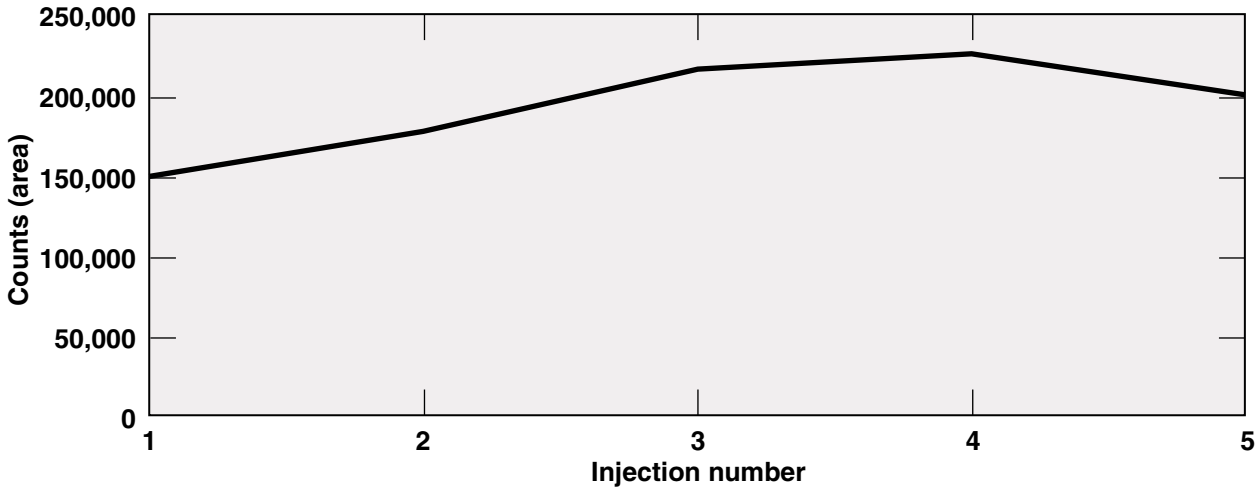

Figure C-1. Sequential injections of Amiton (50 ppm) showing "seasoning" of the chromatographic column.

should be made, and the column thermally purged for about 20 minutes at 300°C after each injection, prior to analyzing additional samples. Such procedure eliminates any small traces of organophosphorous compound retained on the column. Thermal purging is only necessary if the analysis to be performed involves trace-level materials.

Prior to sample analysis, calibration curves of each of the surrogates dissolved in methylene chloride were run at approximately 5, 10, 20, and 50 ppm. The results are shown in Figures C-2 through C-4.

Results for the oxidation of CEES are shown in Table C-1. Results for the oxidation of Amiton are shown in Table C-2.

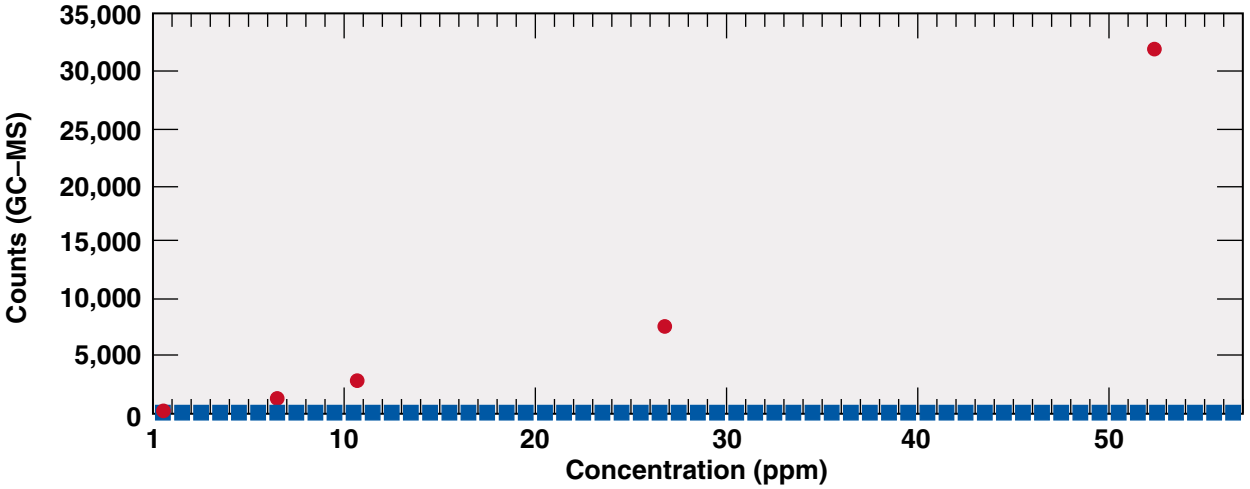

Figure C-2. Calibration curve for Amiton.

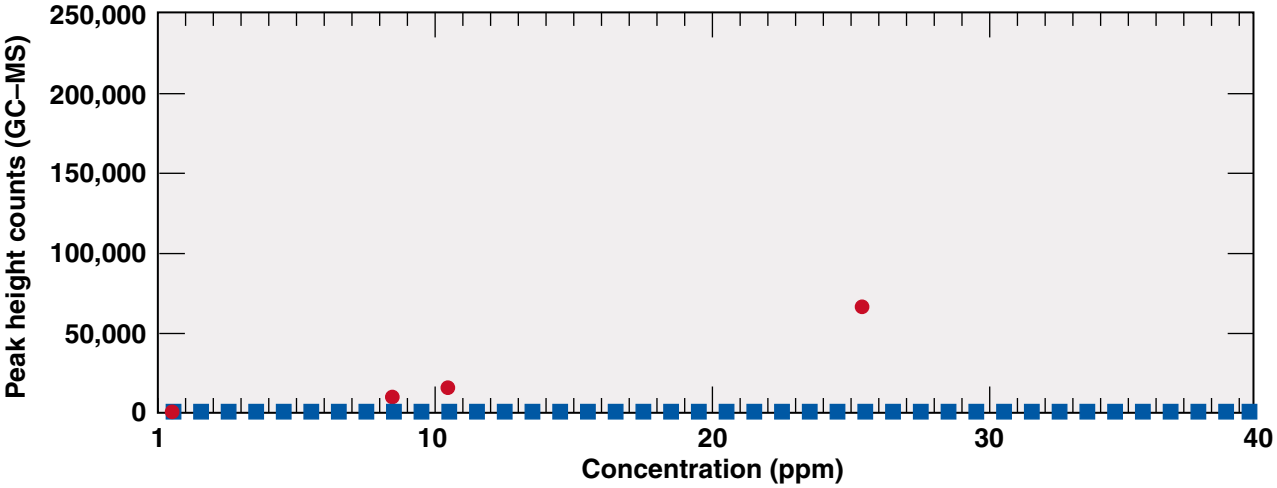

Figure C-3. Calibration curve for diphenyl chlorophosphate (DPCP).

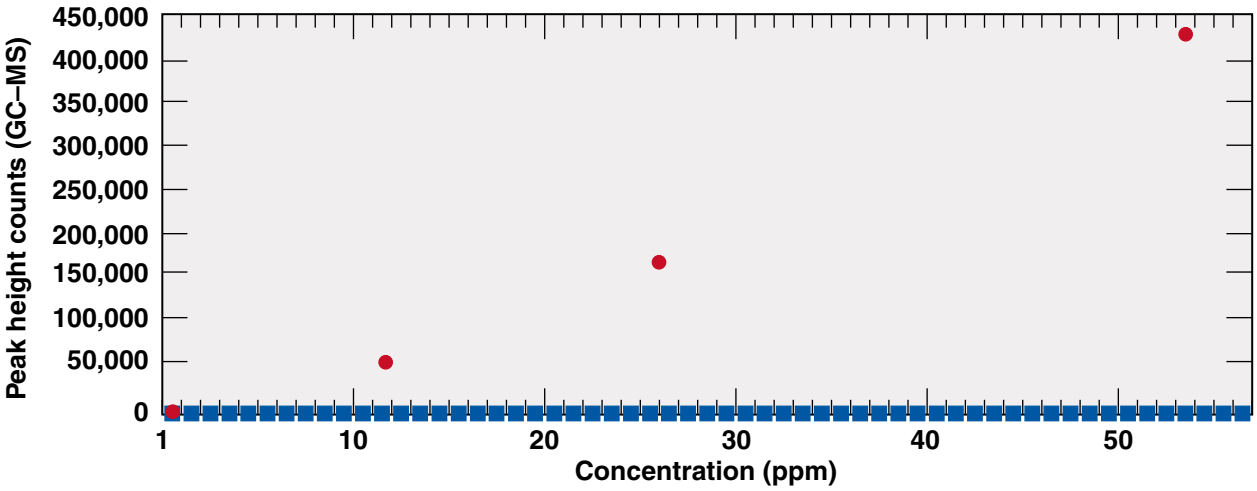

Figure C-4. Calibration curve for chloroethyl ethylsulfide (CEES).

Table C-1. Oxidation of chloroethyl ethylsulfide (CEES).

Oxidizer [a,b,c]	Reaction time (min)	Percent oxidized
Sodium hypochlorite	30	91
H_2O_2 (Fenton's reagent)	30	100
Ammonium peroxydisulfate	10	40
	30	100
Potassium peroxymonosulfate (Oxone)	10	100
	30	100

[a] All reactions were performed at a pH of 3, except for the sodium hypochlorite at pH = 12.
[b] The ratio of oxidizer to CEES was 2.
[c] Oxidations, except for sodium hypochlorite, were catalyzed by 10 ppm Cu^{2+}.

Table C-2. Oxidation of Amiton.

Oxidizer [a,b,c]	Reaction time (min)	Percent oxidized
Sodium hypochlorite	30	30
H_2O_2 (Fenton's reagent)	30	0
Ammonium peroxydisulfate	10	20
	30	90
Potassium peroxymonosulfate (Oxone)	10	20
	30	93
	40	99+

[a] All reactions were performed at a pH of 3, except for the sodium hypochlorite at pH = 12.
[b] The ratio of oxidizer to CEES was 2.
[c] Oxidations, except for sodium hypochlorite, were catalyzed by 10 ppm Cu^{2+}.

Decomposition of DPCP

In preparation for decomposition experiments with DPCP, it was noted that the value (in counts) for the 50-ppm DPCP standard had decreased by about a factor of 10; i.e., when originally measured in January 1998, the 50-ppm standard gave on the order of 10^6 counts, versus 10^5 counts when measured in August 1998. A small portion of the stock of DPCP had been transferred to a separate vial and had been used in the more recent measurements. When we remeasured the stock material, the value for a 50-ppm solution of DPCP was, in fact, 10^6 counts.

Our preliminary interpretation of these data is that the hydrolysis of DPCP is catalyzed by the glass walls of the vessel. (The small vial into which the DPCP had been transferred was not silanized or otherwise treated.) Additional evidence to support this interpretation is found in the results of experiments in which DPCP was reacted with Oxone solution. In the presence of glass filter material, the DPCP is completely decomposed, whereas, without the presence of the filter, little or no decomposition occurs.

Effect of Copper Ion

Preliminary work at LLNL and extensive experimentation at Los Alamos National Laboratory showed that Cu^{2+} is itself moderately effective in killing BG spores. In subsequent tests, Cu^{2+} was added to the Oxone solution for the purpose of catalyzing the hydrolysis of G agents and to add some free radical nature to the oxidation (pseudo Fenton chemistry) of the other agents. We had already noted that the rate of hydrolysis of the G surrogate, DPCP, was not affected by the presence of Cu^{2+}. Additional experiments were performed on CEES and Amiton without adding the copper ion. The results of these experiments show that there is no significant decrease in the decomposition of these materials within 30 min by Oxone solution in the absence of Cu^{2+} (see Figure C-5). We found that Cu^{2+} did not enhance the effectiveness of Oxone against either chemical or biological organisms.

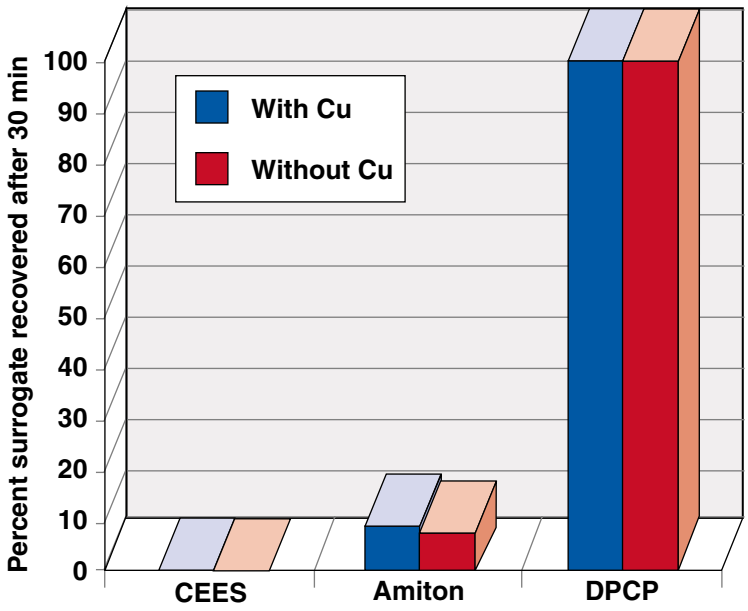

Figure C-5. Effect of copper ion on reaction of Oxone with CW surrogates.

Initial Lab Screening for Oxidation of Biological Agent Simulants

The initial oxidation experiments were carried out using the following general procedure:

- BG spore culture was diluted to approximately 5×10^8 cells/mL.
- Aliquot 1 mL of spores into seven Eppendorf tubes.
- Spin tubes 10 to 15 sec; discard supernatant.
- Resuspend pellets in 1.5 mL of the following chemicals at the following concentrations (procedures varied as a function of oxidizer tested).

Test tube number	Oxidizing reagent normality	Diluent addition (water) (µL)	Oxidizing reagent addition (µL)	Catalyst addition (µL)	Oxidizing reagent final normality
1	1.5	485	1000	0 to 15	1
2	1.0	485	1000	0 to 15	0.67
3	0.45	485	1000	0 to 15	0.3
4	0.15	485	1000	0 to 15	0.1
5	0.015	485	1000	0 to 15	0.01
6 H_2SO_4	0	1485	1000	0 to 15	0
7 H_2O blank	0	1500	1000	0	0

- Let tubes sit at room temperature for 30 min.
- Spin cells in microfuge for a few seconds; remove supernatant.
- Rinse cells in 1 mL of appropriate media two times.
- Resuspend cells in 1 mL fresh nutrient broth.
- Prepare serial dilutions of each pellet using appropriate media as diluent.
- Plate 100 µL of each diluent onto a plate in duplicate.
- Count number of colonies per plate.

Additional Oxidation of Biological Agent Simulants

The effect of a subset of the oxidative decontaminants was determined on two biological agent simulants: *Pantoea herbicola*, a simulant for bacterial agents, and BG spores, a simulant for anthrax.

The general protocol for BW agent testing at LLNL was a follows:

- 0.1- to 1.0-mL suspensions of approximately 5×10^8 cells/mL were used.
- Increasing concentrations (usually 5 to 7) of the oxidants to be tested were prepared.
- Cells or spores were resuspended in the oxidants for 30 minutes.
- Reaction was quenched, and material was washed three times with potassium phosphate buffer.
- Cells or spores were plated on nutrient agar and incubated at 30°C for 24 hours.
- Colonies were visually counted.

Results of the two oxidizers, peroxydisulfate and hypochlorite, tested against biological agent simulants are shown in Figure C-6.

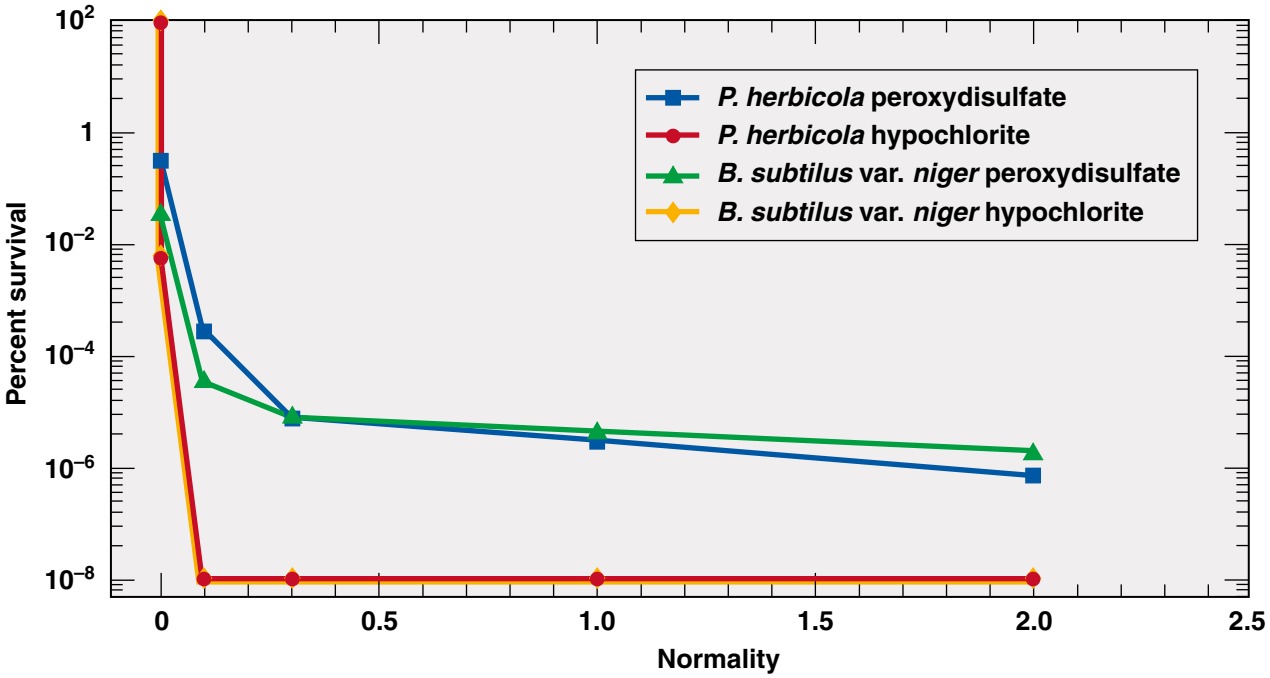

Figure C-6. Effect of oxidizers peroxydisulfate and hypochlorite on vegetative bacterial cells after 30-min exposures.

Figure C-7 shows the results of gel electrophoresis following oxidation/deactivation of ovalbumin by 0.01 N Oxone, compared to two concentrations of hydrogen peroxide (5 and 15% standards), and water alone. The tests were conducted in 12% SDS polyacrylamide gel, and markers appear on the left and right sides. Proteins migrate from the top to the bottom of the image shown, and oxidation slows the migration. The data support the effectiveness of Oxone against ovalbumin.

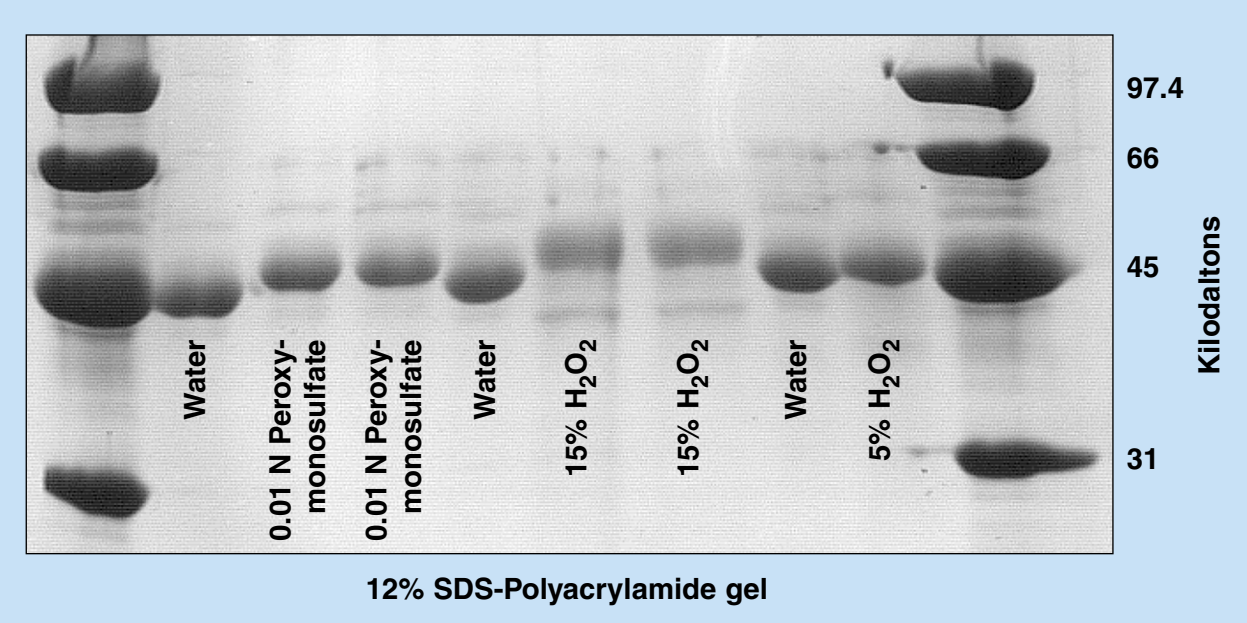

Figure C-7. Gel electrophoresis of ovalbumin following oxidation by Oxone.

Appendix D: Testing Against VX and GD in the Czech Republic

L-Gel-115 was tested against the chemical agents VX and GD in the Czech Republic as a part of a series of tests of chemical agent detectors and chemical agent decontaminants. The tests were performed in October of 1998 in and outdoor arena on concrete and asphalt.

The agent was deposited using a hand sprayer at an areal density of approximately $15g/m^2$. Whereas a large surface area was contaminated, approximately 20 m^2, the analytical results were obtained from a set of smaller samples that were 25 cm^2 in area. The experiments were performed as follows.

1. The agent was sprayed on a circular area of about 20 m^2. Samples to be analyzed were at the outer circumference of the circular area.

2. A set of samples was removed immediately after deposition for chemical analysis to determine the actual spray density.

3. A period of approximately 2 hours elapsed while experiments were performed using four different detection systems.

4. A second set of samples was collected for analysis to determine the amount of agent present at the time of decontamination.

5. Four decontaminants were applied on quadrants of the circular area, one decontaminant to each area. Thus, each decontaminant was sprayed on approximately 5 m^2 of contaminated surface.

6. The decontaminants were allowed to remain in contact for 30 minutes before samples were collected for analysis.

7. Sample substrates were aged concrete, new concrete, aged asphalt, and new asphalt. Aged materials were more than 20 years old. The VX was only tested on new materials.

8. Samples were analyzed for remaining activity.

Upon receipt in the laboratory, samples were washed with distilled water. Each sample was then placed in a stainless-steel cuvette and extracted for 15 min. with isopropanol in an ultrasonic bath. An aliquot of the isopropanol was then analyzed by a spectroscopic technique designed to detect residual anticholinergic activity.

The spectrometric method involves the chemical reaction of the analyte in a process that results in a "colored" species whose concentration is proportionally related to the concentration of analyte and can be determined by measuring its optical density at a given wavelength.

Determination of nerve agents is done by reacting the analyte agent with horse serum choline esterase. The inhibited enzyme is then reacted with butyrylthiocholine iodide (BTCI) to hydrolyze BTCI to thiocholine. The thiocholine is then reacted with Ellmanov reagent (bis 4-nitro-5-carboxyphenyl disulfide). The concentration of adduct of thiocholine and Ellmanov reagent is measured spectrometrically. The log of the concentration of agent is related inversely to the log of the absorbance by a complex fourth-order polynomial. Note the inverse relationship; i.e., the higher the absorbance, the lower the concentration of agent. This method for determining nerve agents is not completely specific in that there are interfering reactions from any material that has anticholinergic activity and from any material that reacts with Ellmanov reagent, whether it is toxic or not. In either case, quantification of the analyte of interest cannot be accomplished unless any additional components can be identified and quantified.

In addition, the effectiveness of the extraction procedure is not known. However, because all samples were handled in exactly the same manner, the results should be valid on a comparison basis.

Appendix E: Testing Against Real Agents at Edgewood Forensic Center

LLNL contracted with the Edgewood Chemical and Biological Forensic Analytical Center at the Edgewood Chemical Biological Center (ECBC), Aberdeen Proving Ground, MD, to perform a series of tests of L-Gel-115 decontaminant against real chemical agents. Sample substrates were the same polyurethane painted oak, acrylic painted steel, and indoor-outdoor carpet materials that were used in surrogate testing at LLNL. The agents selected were pinacolylmethylphosphono fluoridate (GD), O-ethyl S-[2-(diisopropylamino)ethyl]methylphosphono thioate (VX), and bis (2-chloroethyl) sulfide (HD or Sulfur mustard).

Experiments were carried out by placing 2.5 µL of agent on the sample substrate and allowing it to stand for 15 minutes. A 1.0-g aliquot of L-Gel was placed on the sample and allowed to dry for about 24 hours. The entire sample was then extracted with 10 mL of methylene chloride and analyzed by gas chromatography (GC) and flame photometric detector (FPD), the latter for sulfur and phosphorous detection. The experiments were performed in triplicate. The estimated detection limits are:

- GD = 0.1 µg/mL.
- HD = 1.0 µg/mL.
- VX = 0.1 µg/mL.

Table E-1 summarizes the results.

Table E-1. Amount of agent recovered from three substrates following decontamination.

Substrate/agent	GD (%)	HD	VX
Painted oak	6.01 ± 2.01	BLD [a]	BLD
Painted metal	21.2 ± 11.0	BLD	BLD
Carpet	1.66 ± 1.0	BLD	BLD

[a] BLD = below level of detection.

The experiments with GD were repeated approximately five months later on painted surfaces and on unpainted metal. Just prior to the addition of L-Gel, a small amount (10%) of methylethyl ketone (MEK) was added as a co-solvent. The experiments then proceeded as before. Table E-2 summarizes the results.

Table E-2. Amount of GD recovered from various substrates following decontamination.

Substrate	GD without co-solvent (%)	GD with co-solvent (%)
Painted oak	6.01 ± 2.01	4.31 ± 1.27
Painted metal	21.2 ± 11.0	11.4 ± 0.8
Unpainted metal	—	2.42 ± 1.36

These results show that addition of a co-solvent enhances the destruction of GD even though the L-Gel was 5 months old at the time of testing, and we had previously shown that L-Gel reacts with MEK. The results also indicate that the problem is the solubility of GD in paint.

Appendix F: Modified LLNL Procedure for Surrogate BW Agent Field Testing

This appendix provides the procedures used to test decontamination reagents or carrier systems for biological incidents in civilian and public sectors. It features the methods for sampling and recovery of BW agent simulant contamination on several material surfaces tested at the U.S. Army Dugway Proving Ground, West Desert Test Center, UT, in December 1999.

Surrogate Spore-Forming Bacterium

The surrogate organism used during the test was the spore-forming bacterium *Bacillus subtilis* var. *niger*, also known as *Bacillus globigii* (BG). This bacterium closely simulates *Bacillus anthracis*; however, it may be somewhat easier to kill than *B. anthracis*. BG is a gram positive, spore-forming, durable spore that is common in certain soils, noninfectious, easily grown in culture, easily detected, and meets Biosafety Level 1. BG in trypticase soy agar grows into a distinctive colony that is easily identified visually. The BG solution used to contaminate the test materials was produced from a dry powder (10^{11} spores/mg) that was suspended in a 1:100 buffer solution.

Spore Dissemination and Contamination

BG was applied to test panels using a commercial airbrush. A Badger Airbrush 100CL containing the BG slurry was directed from a distance of about 0.46 m (18 in.) onto each test panel. The nozzle sprayed a fine mist of the slurry perpendicular to the surface of vertically suspended panels. The target value for BG deposition densities was in the range of 10^8 to 10^9 colony forming units (cfu) per sample area (Larson and Harper, 1999).

BG spore counts from panels contaminated in Trials 1 through 4, prior to the decontamination application, ranged from 10^4 to 10^5 average cfu/mL. The average cfu/mL was multiplied by the dilution factor [each swab in 20-mL phosphate buffered saline (PBS)] so that the average initial spore population found in a 10.16-cm^2 area of the test panels was between 10^5 and 10^7 cfu.

Test Panels and Sample Area

The size of the sampled area was 10.16 cm^2 (4 $in.^2$). Three different areas for each panel were sampled. The panels consisted of:

- Unused acoustic ceiling tile.
- Commercial carpet, tightly woven.
- Fabric covered office partition panels.
- Smooth, painted wallboard.
- Concrete block slab.
- CARC-painted metal.

Sterile, polyfiber-tipped swabs were rolled back and forth within the 4-in.^{2} (2-in. × 2-in.) area and placed in a test tube containing 20 mL of PBS containing 100-mM sodium thiosulfate and 0.1% TritonX100. The test tubes containing the swab samples were stored at 4°C until they were processed 24 to 48 hr later.

Standardized Negative Control

Controls were run with manipulations similar to those for the treated panel sets used for decontamination. Five percent of the total number of samples were method blanks (1:20 samples). Paraformaldehyde is the industrial standard for sterilization. As a control for comparison of candidate decontamination treatments, a series of contaminated panels were treated with paraformaldehyde (Larson and Harper, 1999). The contaminated item was placed inside a sealed room or container. High humidity was maintained, and the weight of paraformaldehyde that was vaporized was determined by the total volume of the space.

If contamination was noticed on the paraformaldehyde-treated panel samples, it was assumed that the contamination took place after the decontamination agent was applied, and the number of cfu was subtracted from the treated sample cfu results.

Colony Counting Criteria

Plates were counted manually. All plates that had 300 or fewer colonies were counted. Plate samples were prepared to give from 30 to 300 colonies/plate. The aim was to have at least one dilution giving colony counts between these limits. If the total number of colonies was less than 30 from undiluted sample, the 30 to 300 rule was disregarded, and the results 0 to 30 were recorded according to *Standard Methods* (Clesceri, Greenberg, and Trussell, 1989). With this exception, only plates having 30 to 300 colonies were considered in determining the plate count. The bacterial counts per milliliter were computed by multiplying the average number of colonies per plate by the reciprocal of the dilution used. Results were reported as cfu per milliliter. If plates from all dilutions of any sample had no colonies, the count was reported as less than one (<1) times the reciprocal of the corresponding lowest dilution. If the number of colonies per plate exceeded 300, the results reported followed the rules for estimation sited in heterotrophic plate count (9215) of the *Standard Methods.*

Media

The composition per liter of Trypticase™ Soy Agar (TSA; ATCC Medium 18) was as follows:

- Pancreatic digest of casein 17.0 g.
- Agar 15.0 g.
- NaCl 5.0 g.
- Papaic digest of soybean meal 3.0 g.

- K_2HPO_4 2.5 g.
- Glucose 2.5 g.
- 1 L deionized H_20.

The agar was autoclaved for 20 minutes at 121°C (15 psi). The cooled (55°C) agar was poured into Petri dishes at a pH of 7.3 at 25°C (Atlas and Parks, 1993).

The swab solution (PBS with sodium thiosulfate and TritonX100) was made in 40-L batches using 4-L of 10X PBS (Sigma), 632 g of sodium thiosulfate (100 mM), 40-mL TritonX100 (0.1%), and 36-L sterile water. This solution was sterile filtered through a 0.45-µm filter.

Experimental Procedure

1. Each type of panel—acoustic ceiling tile, commercial carpet (tightly woven), fabric covered office partition panels, smooth painted wallboard, concrete block slab, and painted metal—was contaminated with BG. Each sample was 0.41 m × 0.41 m (16 in. × 16 in.). All panels, except the cement block, were placed in a vertical position (Larson and Harper, 1999).

2. Panels prepared as in Step 1 above were treated in various ways with decontaminating solutions and gases, usually in triplicate. The detailed treatment depended on the decontaminating reagent used. The decontaminating agent was applied between 1100 and 1400 hours and left overnight.

3. Technicians swabbed the treated panels starting at 0800 hours. Therefore, the agent application ranged between 18 to 21 hours at room temperature. The ambient temperature ranged from 24° to 65°F during the test. Each panel was sampled using swabs at three random locations on the panel to determine the baseline of initial contamination.

4. The decontamination reaction was quenched by dropping the swab into a culture tube with 20-ml PBS containing 0.1 TritonX100 and 100-mM sodium thiosulfate. Corresponding blanks containing swab solutions served as controls. The addition of TritonX100 to the buffer significantly decreases spore clumping. The thiosulfate neutralized the hypochlorite moieties, which could affect bacterial growth. To extract spores from the swabs, the samples were placed on a Wrist Action shaker (Burrell Scientific Company, model 75) and shaken for 10 minutes.

5. Each swab sample was labeled with pertinent information and bar coded. The bar code followed the samples through all sample processes.

6. The spore suspension was serially diluted in a sequence between 10^0 to 10^6. This dilution was taken into account when calculating the bacterial population.

7. A volume of 0.2 mL of diluent was delivered to each plate and spread using standard techniques. The spore population was quantified by culturing, in triplicate, on TSA (see *Assay Procedures*, DTC SOP 70-100, MT-L389).

8. The plates were incubated at 37°C for 24 hours, then counted visually by trained personnel. The average number of bacterial colonies and the standard deviations were determined.

Appendix G: Operating Procedures for Application of L-Gel

Information in Appendix G supports the use and application of L-Gel-115 and L-Gel-200, which are specially formulated gelled decontamination materials that can be sprayed using commercially available equipment. (The L-Gel 200 formulation is specifically designed for the decontamination of thickened CW agents.) The gel is designed to adhere to vertical surfaces and the undersides of horizontal surfaces.

The active ingredient of the gel is potassium peroxymonosulfate. Peroxymonosulfate is an oxidative compound. A 0.5- to 0.8-N solution of peroxymonosulfate is gelled using fumed silica. Inorganic silica is compatible with strong oxidizing agents, such as peroxymonosulfate. The silica forms a thixotropic gel, that is, under shear forces it has very low viscosity, but when there is no shear, it becomes very viscous. Thus, it can be sprayed using an atomizing nozzle, such as a paint sprayer. For indoor use, after the gel is dry, it can simply be vacuumed up and discarded. For outdoor use, no cleanup is required.

General Safety Concerns

Because the peroxymonosulfate in L-Gel is an oxidative, acidic substance, prolonged contact with skin and clothes should be avoided. If contact occurs, wash with water. Avoid contact with eyes, and if contact occurs flush immediately with water. Latex gloves, standard goggles, and a laboratory coat or coveralls should provide adequate protection. Direct inhalation during panel treatment should be avoided. L-Gel application should be conducted in a well ventilated area. If panel treatment is conducted indoors, use of a particulate face mask (painter's mask) is recommended. The commercial paint sprayer delivers a moderate pressure stream that can pierce the skin and underlying tissue. Thus, care must be taken never to aim the gun at any part of the body. A fire extinguisher should be present during application. Follow all manufacturer-recommended safety procedures when using commercial spray equipment.

Material Storage Requirements

Premixed L-Gel material should be stored indoors in an air conditioned warehouse-like facility or a typical office building. Unused material should be capped tightly for storage.

Standard Operating Procedures

Equipment and Materials

- Premixed L-Gel material.
- Graco Electric Airless Paint Sprayer (Model XR7 on wheels), or equivalent. Stainless steel nozzles are recommended.
- Cordless rechargeable Dewalt ⅜-in. power drill, or equivalent.

- Stainless steel 5-gallon Squirrel mixer paddles.
- Sprayer extension rod for ceilings and high areas.
- Three-wire, 30-ft extension cord.
- Wagoner All Guard Solution for cleaning.
- Two extra 5-gallon paint buckets.

Power and Water Requirements

- No unusual power or water sources are required.
- Paint sprayer requires a nominal 120-volt circuit and a 3-slot receptacle.
- The sprayer has a grounding plug. A three-wire extension cord that has a 3-blade grounding plug and a 3-slot receptacle should be used.
- Water is needed for cleaning the paint sprayer after use.

Safety Equipment

- Laboratory coats or coveralls.
- Latex gloves.
- Water.
- Standard laboratory goggles.
- Particulate face masks (face shield for splash).

Operating Procedure

1. Assemble and set up Graco paint sprayer (or similar) according to manufacturer's directions.
2. Follow the paint sprayer manufacturer's operating procedures. Adjust the sprayer with water. The nozzle is turned so that the spray fan is vertical.
3. Adjust the nozzle to obtain a spray fan about 1 ft wide when sprayed from about a 3- to 5-ft distance. The spray fan should be just wide enough to provide adequate coating, but not so narrow as to result in substantial runoff.
4. Open the 5-gallon container of L-Gel material and stir with Dewalt drill and Squirrel mixer attachment at low speed until all gel becomes fluid (2 to 3 minutes). (Smaller 1-L bottles of L-Gel can be manually shaken or stirred for about 3 minutes to liquefy the material.)

5. Place or hook the 5-gallon bucket correctly with respect to the paint sprayer for mobility.

6. Cover the area to be decontaminated with a single coat before going over any portion more than once. Repeat three times allowing 1 to 2 minutes between applications of the L-Gel material so that the material can become tacky.

7. The coating is applied at a rate of about 1 square meter in 5 to 7 seconds. Each coating should be applied to at least 5-mil thickness or until the surface is clearly wetted and material starts to run.

8. Spraying should be continuous to avoid nozzle plugging. When the sprayer is not in use, maintain the "on" position and spray directly into a reservoir bucket.

9. Contact time required for the L-Gel to maximize effectiveness is one hour.

10. Clean the sprayer by spraying water through it. Cleaning the containers with water. Wagoner All Guard Cleaner should be used per manufacturer's directions.

11. If necessary, when indoors, sweep up any dried particles of L-Gel and dispose.

12. Conduct sampling and analysis activities as appropriate.